BEST AVAILABLE IMAGES

Defective images within this document are accurate representation of The original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents *will not* correct images, please do not report the images to the Image Problem Mailbox.

Access DB# 96214

SEARCH REQUEST FORM

Scientific and Technical Information Center

MAR 28 2003

Requester's Full Name: [illegible] C. Examiner #: 7124 Date: 28 MAR 03

Art Unit: 164 Phone Number 30[illegible] Serial Number: 09/998,625

Mail Box and Bldg/Room Location: 2007, CM1 Results Format Preferred (circle): PAPER DISK E-MAIL

2007, CM1

If more than one search is submitted, please prioritize searches in order of need.

****

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: see attached sheet

Inventors (please provide full names):

Earliest Priority Filing Date: 02 DEC 1997

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

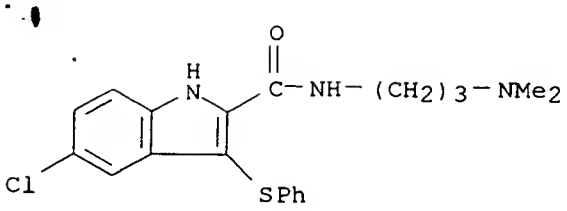
Please search claims 20 and 21 and 25

Point of Contact:
Barb O'Bryen
Technical Information Specialist
STIC CM1 6A05 308-4291

****

STAFF USE ONLY	Type of Search	Vendors and cost where applicable
Searcher: [illegible]	NA Sequence (#)	STN 693
Searcher Phone #:	AA Sequence (#)	Dialog
Searcher Location:	Structure (#) 4	Questel/Orbit
Date Searcher Picked Up: 4-9-03	Bibliographic	Dr. Link
Date Completed: 4-9-03	Litigation	Lexis/Nexis
Searcher Prep & Review Time:	Fulltext	Sequence Systems
Clerical Prep Time:	Patent Family	WWW/Internet
Online Time: 168		Other (Specify)

PTO-1590 (8-01)

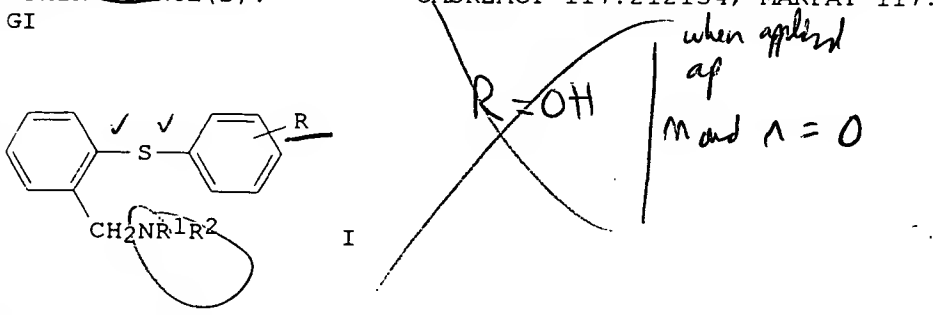


● HCl

L55 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1992:612134 CAPLUS
DOCUMENT NUMBER: 117:212134
TITLE: Preparation of new antimicrobial (phenylthio)benzylamines
INVENTOR(S): Jilek, Jiri; Sindelar, Karel; Kmonicek, Vojtech; Pomykacek, Josef; Hola, Vladislava; Protiva, Miroslav
PATENT ASSIGNEE(S): Czech.
SOURCE: Czech., 10 pp.
CODEN: CZXXA9
DOCUMENT TYPE: Patent
LANGUAGE: Czech
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 272944	B1	19910312	CS 1989-1456	19890308

OTHER SOURCE(S): CASREACT 117:212134; MARPAT 117:212134
GI

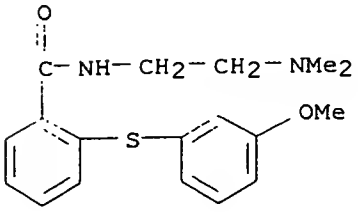


AB The title compds. (I; R1 = R2 = H, Et, Pr, Me2CH; R1 = Me2NCH2CH2, R2 = H, Me) [II; R = (OH)n; n = 1, 2] and their salts, were prepd. by demethylation of the parent anisoles II (R = (OMe)n; n as above)] by heating with pyridine-HCl or 48% HBr; or by BBr3 at the ambient temp., followed by neutralization of the resulting bases. Thus, 2-(2-methoxyphenylthio)benzoic acid was converted (93%) to its chloride, then amidated (95%) by aq. NH3, and the amide reduced (77%, isolated as the HCl salt) by LiAlH4 in Et2O. The resulting 2-[(2-methoxyphenyl)thio]benzylamine (5.8 g) was stirred and heated at 210-215.degree. with 14 g pyridine-HCl to give 4.1 g title compd. I (R = 2-HO, R1 = R2 = H) (III). The latter had IC50 = 50 mg/L against Pseudomonas aeruginosa, Proteus vulgaris, and Trichophyton mentagrophytes. Approx. 17 I were prepd. and several I (tested as HCl- or maleate salts) had IC50 of 16-128 mg/L in growth inhibition tests with 7 microorganisms. I in mice had oral acute toxicity LD50 of 146-704 mg/kg.

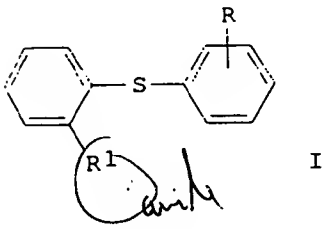
IT **127906-90-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
Searched by Barb O'Bryen, STIC 308-4291

(prepn. and reaction of, in prepn. of antimicrobial agent)
RN 127906-90-5 CAPLUS
CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[(3-methoxyphenyl)thio]- (9CI) (CA INDEX NAME)

C2



L55 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1990:458596 CAPLUS
DOCUMENT NUMBER: 113:58596
TITLE: Potential antidepressants: 2-(methoxy- and hydroxyphenylthio)benzylamines as selective inhibitors of 5-hydroxytryptamine re-uptake in the brain
AUTHOR(S): Jilek, Jiri; Sindelar, Karel; Pomykacek, Josef; Kmonicek, Vojtech; Sedivy, Zdenek; Hrubantova, Marta; Holubek, Jiri; Svatek, Emil; Ryska, Miroslav; et al.
CORPORATE SOURCE: Res. Inst. Pharm. Biochem., Prague, 130 60, Czech.
SOURCE: Collect. Czech. Chem. Commun. (1989), 54(12), 3294-338
CODEN: CCCCAK; ISSN: 0010-0765
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 113:58596
GI



I

$CONH_2$ $CONHMe$

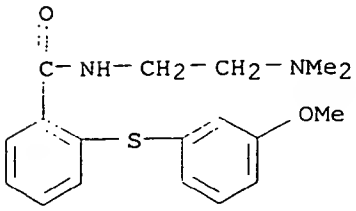
AB 2-, 3-, And 4-methoxythiophenol, and 2,4-, 2,5- and 3,4-dimethoxythiophenol were transformed in two steps to (phenylthio)benzoyl chlorides I (R = 2-, 3-, 4-OMe, 2,4-, 2,5-, 3,4-(OMe)2, R1 = COCl), which were reacted with NH3, MeNH2, Me2NH, Et2NH, Pr2NH, and (Me2CH)2NH to give the amides I [R1 = CONH2, CONHMe, CONMe2CONEt2, CONPr2, CON(CHMe2)2]. These were reduced mostly with LiAlH4 to the amines I (R1 = CH2NH2, CH2NMe2 etc.). These methoxylated amines were demethylated either by heating with pyridine hydrochloride or by treatment with BBr3. Some of the -(methoxy- and hydroxyphenylthio)benzylamines prepd., indicated properties of potential antidepressants being highly active and selective inhibitors of 5-hydroxytryptamine re-uptake in the brain structures and having the typical antireserpine activity. The most interesting compd. of the series is I (R = 3-OH, R1 = CH2NMe2) which is undergoing preclin. studies.

IT **127906-90-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and hydride redn. of)

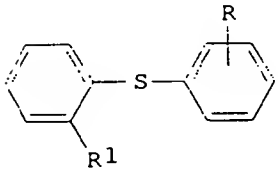
RN 127906-90-5 CAPLUS

Searched by Barb O'Bryen, STIC 308-4291

(prepn. and reaction of, in prepn. of antimicrobial agent)
RN 127906-90-5 CAPLUS
CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[(3-methoxyphenyl)thio]- (9CI) (CA INDEX NAME)



L55 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1990:458596 CAPLUS
DOCUMENT NUMBER: 113:58596
TITLE: Potential antidepressants: 2-(methoxy- and hydroxyphenylthio)benzylamines as selective inhibitors of 5-hydroxytryptamine re-uptake in the brain
AUTHOR(S): Jilek, Jiri; Sindelar, Karel; Pomykacek, Josef; Kmonicek, Vojtech; Sedivy, Zdenek; Hrubantova, Marta; Holubek, Jiri; Svatek, Emil; Ryska, Miroslav; et al.
CORPORATE SOURCE: Res. Inst. Pharm. Biochem., Prague, 130 60, Czech.
SOURCE: Collect. Czech. Chem. Commun. (1989), 54(12), 3294-338
CODEN: CCCCAK; ISSN: 0010-0765
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 113:58596
GI



I

AB 2-, 3-, And 4-methoxythiophenol, and 2,4-, 2,5- and 3,4-dimethoxythiophenol were transformed in two steps to (phenylthio)benzoyl chlorides I (R = 2-, 3-, 4-OMe, 2,4-, 2,5-, 3,4-(OMe)2, R1 = COCl), which were reacted with NH3, MeNH2, Me2NH, Et2NH, Pr2NH, and (Me2CH)2NH to give the amides I [R1 = CONH2, CONHMe, CONMe2CONEt2, CONPr2, CON(CHMe2)2]. These were reduced mostly with LiAlH4 to the amines I (R1 = CH2NH2, CH2NMe2 etc.). These methoxylated amines were demethylated either by heating with pyridine hydrochloride or by treatment with BBr3. Some of the -(methoxy- and hydroxyphenylthio)benzylamines prepd., indicated properties of potential antidepressants being highly active and selective inhibitors of 5-hydroxytryptamine re-uptake in the brain structures and having the typical antireserpine activity. The most interesting compd. of the series is I (R = 3-OH, R1 = CH2NMe2) which is undergoing preclin. studies.
IT **127906-90-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and hydride redn. of)
RN 127906-90-5 CAPLUS

FILE 'CAOLD' ENTERED AT 15:39:31 ON 12 JUN 2000

FILE COVERS 1907-1966
FILE LAST UPDATED: 01 May 1997 (19970501/UP)

This file contains CAS Registry Numbers for easy and accurate substance identification. Title keywords, authors, patent assignees, and patent information, e.g., patent numbers, are now searchable from 1907-1966. TIFF images of CA abstracts printed between 1907-1966 are available in the PAGE display formats.

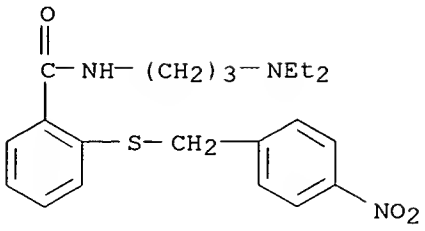
This file supports REG1stRY for direct browsing and searching of all substance data from the REGISTRY file. Enter HELP FIRST for more information.

get

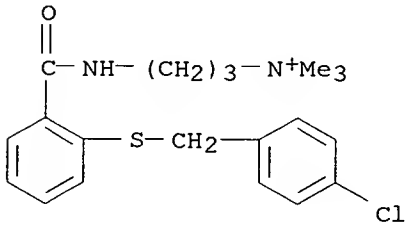
L56 1 L54

=> d iall hitstr l56; fil hom

L56 ANSWER 1 OF 1 CAOLD COPYRIGHT 2000 ACS
ACCESSION NUMBER: CA56:4664g CAOLD
TITLE: dialkylaminoalkylic N-or S-derivs. of 2-mercapto-2,2'-dithio, 2-(alkylthio)-, 2-(aralkylthio)-, and 2-(arylthio)benzamides
AUTHOR NAME: Gialdi, Franco; Ponci, R.; Baruffini, A.
INDEX TERM: 1049-92-9 2634-31-3 2752-93-4 15109-12-3 20904-30-7
32276-24-7 32276-25-8 32276-26-9 72534-70-4 88783-54-4
90793-61-6 90919-33-8 91061-47-1 91430-12-5 91767-36-1
91822-89-8 92199-75-2 92374-01-1 93010-85-6 93994-99-1
94032-03-8 94208-07-8 94262-71-2 94326-49-5
94378-58-2 94437-14-6 94437-53-3
94682-59-4 94758-14-2 94862-94-9 94906-16-8 94907-25-2
94915-86-3 94999-40-3 95277-72-8
95291-17-1 96063-90-0 96067-38-8 96198-56-0
97018-37-6 97393-84-5 97575-12-7 97772-27-5 98051-88-8
98131-92-1 98200-27-2 98397-89-8 98470-98-5 98766-48-4
98883-91-1 98963-55-4 99003-05-1 99729-67-6
100027-88-1 100197-42-0 **100233-06-5**
100321-14-0 103133-24-0 **103193-14-2**
103193-31-3 107305-87-3 107579-58-8 108042-03-1
IT **94378-58-2 94437-14-6 94915-86-3**
94999-40-3 95291-17-1 100027-88-1
100233-06-5 100321-14-0 103193-14-2
RN 94378-58-2 CAOLD
CN Benzamide, o-[(p-chlorobenzyl)thio]-N-[3-(dimethylamino)propyl]- (7CI) (CA INDEX NAME)

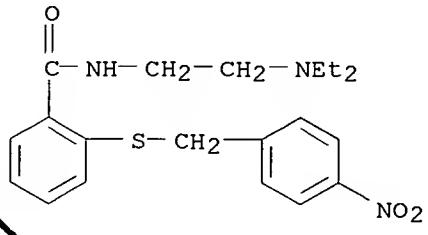


RN 100027-88-1 CAOLD
CN [3-[o-[(p-Chlorobenzyl)thio]benzamido]propyl]trimethylammonium iodide (7CI) (CA INDEX NAME)



● I^-

RN 100233-06-5 CAOLD
CN Benzamide, N-[2-(diethylamino)ethyl]-o-[(p-nitrobenzyl)thio]-, hydrochloride (7CI) (CA INDEX NAME)



● HCl

RN 100321-14-0 CAOLD
CN [3-[o-(Benzylthio)benzamido]propyl]trimethylammonium iodide (7CI) (CA INDEX NAME)

Searched by Barb O'Bryen, STIC 308-4291

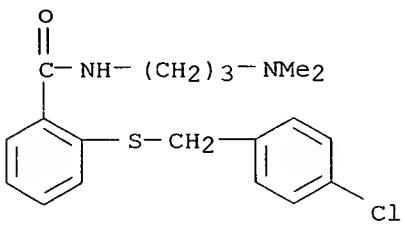
● I^-

RN 103193-14-2 CAOLD
CN Benzamide, o-[(p-chlorobenzyl)thio]-N-[2-(diethylamino)ethyl]-, hydrochloride (7CI) (CA INDEX NAME)

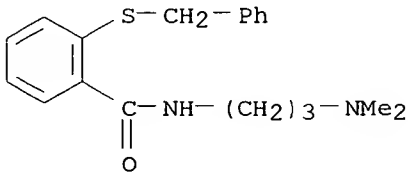
● HCl

FILE 'HOME' ENTERED AT 15:40:50 ON 12 JUN 2000

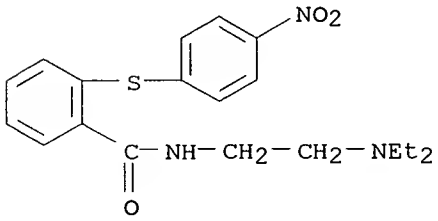
Searched by Barb O'Bryen, STIC 308-4291



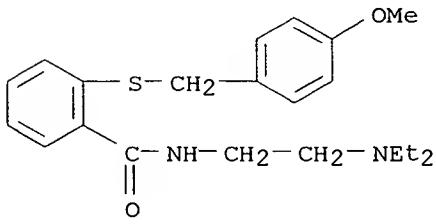
RN 94437-14-6 CAOLD
CN Benzamide, o-(benzylthio)-N-[3-(dimethylamino)propyl]- (7CI) (CA INDEX NAME)



RN 94915-86-3 CAOLD
CN Benzamide, N-[2-(diethylamino)ethyl]-o-[(p-nitrophenyl)thio]- (7CI) (CA INDEX NAME)



RN 94999-40-3 CAOLD
CN Benzamide, N-[2-(diethylamino)ethyl]-o-[(p-methoxybenzyl)thio]- (7CI) (CA INDEX NAME)



RN 95291-17-1 CAOLD
CN Benzamide, N-[3-(diethylamino)propyl]-o-[(p-nitrobenzyl)thio]- (7CI) (CA INDEX NAME)

● HCl

L55 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1992:612134 CAPLUS
DOCUMENT NUMBER: 117:212134
TITLE: Preparation of new antimicrobial (phenylthio)benzylamines
INVENTOR(S): Jilek, Jiri; Sindelar, Karel; Kmonicek, Vojtech; Pomykacek, Josef; Hola, Vladislava; Protiva, Miroslav
PATENT ASSIGNEE(S): Czech.
SOURCE: Czech., 10 pp.
CODEN: CZXXA9
DOCUMENT TYPE: Patent
LANGUAGE: Czech
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 272944	B1	19910212	CS 1989-1456	19890308

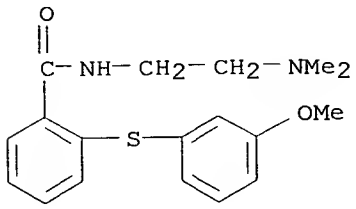
OTHER SOURCE(S): CASREACT 117:212134; MARPAT 117:212134
GI

I

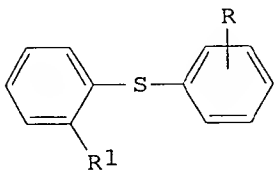
AB The title compds. (I; R1 = R2 = H, Et, Pr, Me2CH; R1 = Me2NCH2CH2, R2 = H, Me) [II; R = (OH)n; n = 1, 2] and their salts, were prepd. by demethylation of the parent anisoles II (R = (OMe)n; n as above)] by heating with pyridine-HCl or 48% HBr, or by BBr3 at the ambient temp., followed by neutralization of the resulting bases. Thus, 2-(2-methoxyphenylthio)benzoic acid was converted (93%) to its chloride, then amidated (95%) by aq. NH3, and the amide reduced (77%, isolated as the HCl salt) by LiAlH4 in Et2O. The resulting 2-[(2-methoxyphenyl)thio]benzylamine (5.8 g) was stirred and heated at 210-215.degree. with 14 g pyridine-HCl to give 4.1 g title compd. I (R = 2-HO, R1 = R2 = H) (III). The latter had IC50 = 50 mg/L against Pseudomonas aeruginosa, Proteus vulgaris, and Trichophyton mentagrophytes. Approx. 17 I were prepd. and several I (tested as HCl- or maleate salts) had IC50 of 16-128 mg/L in growth inhibition tests with 7 microorganisms. I in mice had oral acute toxicity LD50 of 146-704 mg/kg.

IT **127906-90-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)

(prepn. and reaction of, in prepn. of antimicrobial agent)
RN 127906-90-5 CAPLUS
CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[(3-methoxyphenyl)thio]- (9CI) (CA INDEX NAME)



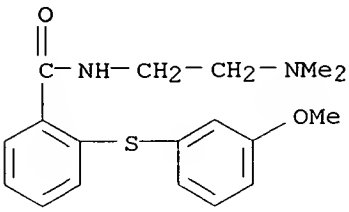
L55 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1990:458596 CAPLUS
DOCUMENT NUMBER: 113:58596
TITLE: Potential antidepressants: 2-(methoxy- and hydroxyphenylthio)benzylamines as selective inhibitors of 5-hydroxytryptamine re-uptake in the brain
AUTHOR(S): Jilek, Jiri; Sindelar, Karel; Pomykacek, Josef; Kmonicek, Vojtech; Sedivy, Zdenek; Hrubantova, Marta; Holubek, Jiri; Svatek, Emil; Ryska, Miroslav; et al.
CORPORATE SOURCE: Res. Inst. Pharm. Biochem., Prague, 130 60, Czech.
SOURCE: Collect. Czech. Chem. Commun. (1989), 54(12), 3294-338
CODEN: CCCCAK; ISSN: 0010-0765
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 113:58596
GI



I

AB 2-, 3-, And 4-methoxythiophenol, and 2,4-, 2,5- and 3,4-dimethoxythiophenol were transformed in two steps to (phenylthio)benzoyl chlorides I (R = 2-, 3-, 4-OMe, 2,4-, 2,5-, 3,4-(OMe)2, R1 = COCl), which were reacted with NH3, MeNH2, Me2NH, Et2NH, Pr2NH, and (Me2CH)2NH to give the amides I [R1 = CONH2, CONHMe, CONMe2CONEt2, CONPr2, CON(CHMe2)2]. These were reduced mostly with LiAlH4 to the amines I (R1 = CH2NH2, CH2NMe2 etc.). These methoxylated amines were demethylated either by heating with pyridine hydrochloride or by treatment with BBr3. Some of the -(methoxy- and hydroxyphenylthio)benzylamines prepd., indicated properties of potential antidepressants being highly active and selective inhibitors of 5-hydroxytryptamine re-uptake in the brain structures and having the typical antireserpine activity. The most interesting compd. of the series is I (R = 3-OH, R1 = CH2NMe2) which is undergoing preclin. studies.
IT **127906-90-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and hydride redn. of)
RN 127906-90-5 CAPLUS

CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[(3-methoxyphenyl)thio]- (9CI)
(CA INDEX NAME)



L55 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1977:552276 CAPLUS
DOCUMENT NUMBER: 87:152276
TITLE: 3-(Heterocyclicthiomethyl)quinoxaline 1,4-dioxides
INVENTOR(S): Urban, Frank J.
PATENT ASSIGNEE(S): Pfizer Inc., USA
SOURCE: U.S., 13 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4038392	A	19770726	US 1975-622057	19751014
NL 7610317	A	19770418	NL 1976-10317	19760916
BE 846532	A1	19770324	BE 1976-1007643	19760924
FR 2327784	A1	19770513	FR 1976-28849	19760924
FR 2327784	B1	19781117		
JP 52048679	A2	19770418	JP 1976-115729	19760927
DE 2645787	A1	19770421	DE 1976-2645787	19761009

PRIORITY APPLN. INFO.: US 1975-622057 19751014
GI For diagram(s), see printed CA Issue.
AB Quinoxaline dioxides I (R = CO2Me, CONH2, substituted carbamoyl, CH2OH, Ac, H; R1 = CH2SR2, CH2SO2R2, CH2SOR2, CH2SO2CH2R2, CH2SO2(CH2)3R2, R2 = N heterocycle) (>100 compds.) were prepd. Thus I (R = CH2OH, R1 = Me) was brominated and treated wtih 1-methyl-2-imidazolethiol to give I (R = CH2OH, R1 = 1-methyl-2-imidazolylthiomethyl), which had min. inhibitory concns. against Streptococcus pyogenes and Escherichia coli 50 and 100 mg/ml.
IT **63205-98-1P 63206-13-3P 63206-17-7P**
63206-29-1P 63206-32-6P 63219-25-0P
64300-90-9P 64300-93-2P 64300-95-4P
RL: BAC (Biological activity or effector, except adverse); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)
(prepn. and bactericidal activity of)
RN 63205-98-1 CAPLUS
CN 2-Quinoxalinecarboxamide, N-[2-(dimethylamino)ethyl]-3-[[(2-pyridinylmethyl)sulfonyl]methyl]-, 1,4-dioxide (9CI) (CA INDEX NAME)

INDEX TF

This file supports REGistRY for direct browsing and searching of all substance data from the REGISTRY file. Enter HELP FIRST for more information.

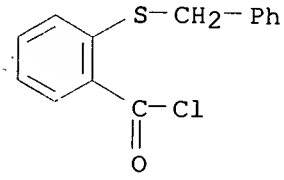
L25 STR
L28 29 SEA FILE=REGISTRY SSS FUL L25
L32 7 SEA FILE=CAOLD ABB=ON L28

=> d iall hitstr l32 1-7; fil hom

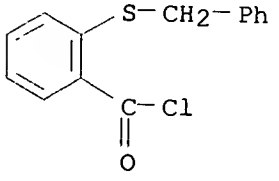
L32 ANSWER 1 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA59:10010f CAOLD
TITLE: 11-(3-dimethylaminopropylidene)-6,11-dihydrodibenz(b,e)thiepin
AUTHOR NAME: Protiva, Miroslav; Rajsner, M.; Votava, Z.; Metysova, J.
DOCUMENT TYPE: Patent

PATENT NO.	KIND	DATE
CZ 105590		

INDEX TERM: 1531-77-7 **1531-81-3** 1531-85-7 96175-10-9
IT **1531-81-3**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L32 ANSWER 2 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA59:2772g CAOLD
TITLE: synthetic ataractics - (VII) 11-(3-dimethylaminopropylidene)-6,11-dihydrodibenzo[b,e]thiepins
AUTHOR NAME: Rajsner, Miroslav; Protiva, M.
INDEX TERM: 113-53-1 897-15-4 1531-77-7 **1531-81-3** 1531-85-7 1699-03-2 1699-04-3 1745-46-6 33301-21-2 34129-26-5 96175-10-9
IT **1531-81-3**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L32 ANSWER 3 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA58:4574c CAOLD
TITLE: synthetic medicinals - (VIII) tricyclic thiazepine and thiepin derivs.
AUTHOR NAME: Gadient, Fulvio; Jucker, E.; Lindenmann, A.; Taeschler, M.

FILE 'CAOLD' ENTERED AT 15:39:31 ON 12 JUN 2000
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

FILE COVERS 1907-1966
FILE LAST UPDATED: 01 May 1997 (19970501/UP)

This file contains CAS Registry Numbers for easy and accurate substance identification. Title keywords, authors, patent assignees, and patent information, e.g., patent numbers, are now searchable from 1907-1966. TIFF images of CA abstracts printed between 1907-1966 are available in the PAGE display formats.

This file supports REGlstRY for direct browsing and searching of all substance data from the REGISTRY file. Enter HELP FIRST for more information.

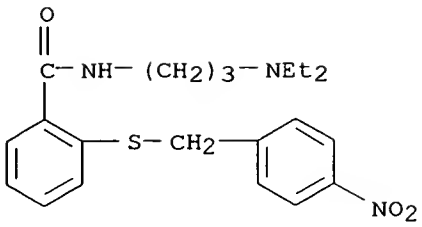
get

L56 1 L54

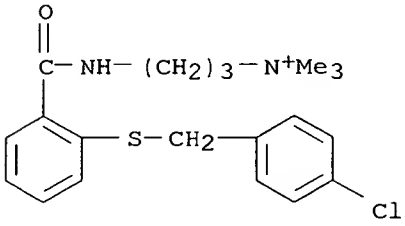
=> d iall hitstr l56; fil hom

L56 ANSWER 1 OF 1 CAOLD COPYRIGHT 2000 ACS
ACCESSION NUMBER: CA56:4664g CAOLD
TITLE: dialkylaminoalkylic N-or S-derivs. of 2-mercapto-2,2'-dithio, 2-(alkylthio)-, 2-(aralkylthio)-, and 2-(arylthio)benzamides
AUTHOR NAME: Gialdi, Franco; Ponci, R.; Baruffini, A.
INDEX TERM: 1049-92-9 2634-31-3 2752-93-4 15109-12-3 20904-30-7
32276-24-7 32276-25-8 32276-26-9 72534-70-4 88783-54-4
90793-61-6 90919-33-8 91061-47-1 91430-12-5 91767-36-1
91822-89-8 92199-75-2 92374-01-1 93010-85-6 93994-99-1
94032-03-8 94208-07-8 94262-71-2 94326-49-5
94378-58-2 **94437-14-6** 94437-53-3
94682-59-4 94758-14-2 94862-94-9 94906-16-8 94907-25-2
94915-86-3 **94999-40-3** 95277-72-8
95291-17-1 96063-90-0 96067-38-8 96198-56-0
97018-37-6 97393-84-5 97575-12-7 97772-27-5 98051-88-8
98131-92-1 98200-27-2 98397-89-8 98470-98-5 98766-48-4
98883-91-1 98963-55-4 99003-05-1 99729-67-6
100027-88-1 100197-42-0 **100233-06-5**
100321-14-0 103133-24-0 **103193-14-2**
103193-31-3 107305-87-3 107579-58-8 108042-03-1
IT **94378-58-2** **94437-14-6** **94915-86-3**
94999-40-3 **95291-17-1** **100027-88-1**
100233-06-5 **100321-14-0** **103193-14-2**
RN 94378-58-2 CAOLD
CN Benzamide, o-[(p-chlorobenzyl)thio]-N-[3-(dimethylamino)propyl]- (7CI) (CA INDEX NAME)

Searched by Barb O'Bryen, STIC 308-4291

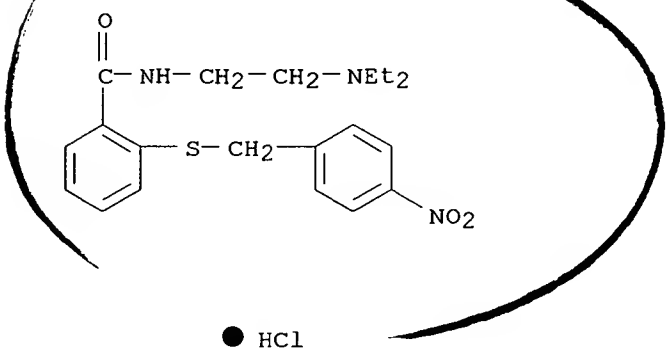


RN 100027-88-1 CAOLD
CN [3-[o-[(p-Chlorobenzyl)thio]benzamido]propyl]trimethylammonium iodide (7CI) (CA INDEX NAME)



● I^-

RN 100233-06-5 CAOLD
CN Benzamide, N-[2-(diethylamino)ethyl]-o-[(p-nitrobenzyl)thio]-, hydrochloride (7CI) (CA INDEX NAME)



● HCl

RN 100321-14-0 CAOLD
CN [3-[o-(Benzylthio)benzamido]propyl]trimethylammonium iodide (7CI) (CA INDEX NAME)

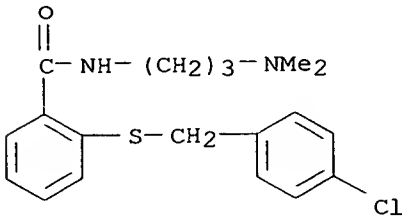
● I^-

RN 103193-14-2 CAOLD
CN Benzamide, o-[(p-chlorobenzyl)thio]-N-[2-(diethylamino)ethyl]-, hydrochloride (7CI) (CA INDEX NAME)

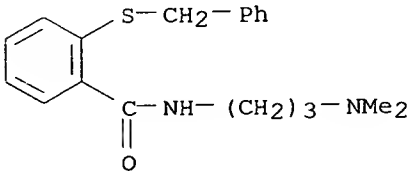
● HCl

FILE 'HOME' ENTERED AT 15:40:50 ON 12 JUN 2000

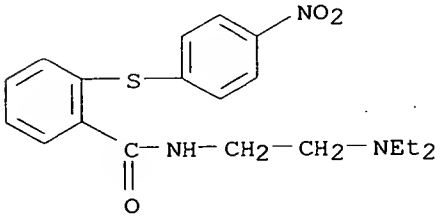
Searched by Barb O'Bryen, STIC 308-4291



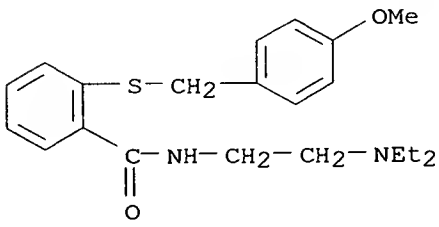
RN 94437-14-6 CAOLD
CN Benzamide, o-(benzylthio)-N-[3-(dimethylamino)propyl]- (7CI) (CA INDEX NAME)



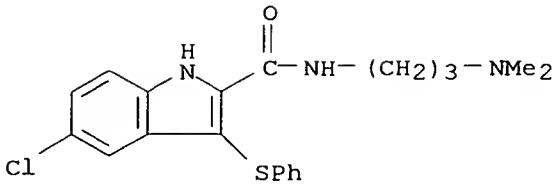
RN 94915-86-3 CAOLD
CN Benzamide, N-[2-(diethylamino)ethyl]-o-[(p-nitrophenyl)thio]- (7CI) (CA INDEX NAME)



RN 94999-40-3 CAOLD
CN Benzamide, N-[2-(diethylamino)ethyl]-o-[(p-methoxybenzyl)thio]- (7CI) (CA INDEX NAME)



RN 95291-17-1 CAOLD
CN Benzamide, N-[3-(diethylamino)propyl]-o-[(p-nitrobenzyl)thio]- (7CI) (CA INDEX NAME)

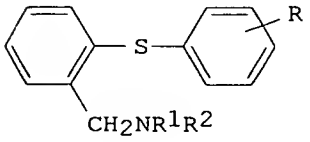


● HCl

L55 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1992:612134 CAPLUS
DOCUMENT NUMBER: 117:212134
TITLE: Preparation of new antimicrobial (phenylthio)benzylamines
INVENTOR(S): Jilek, Jiri; Sindelar, Karel; Kmonicek, Vojtech; Pomykacek, Josef; Hola, Vladislava; Protiva, Miroslav
PATENT ASSIGNEE(S): Czech.
SOURCE: Czech., 10 pp.
CODEN: CZXXA9
DOCUMENT TYPE: Patent
LANGUAGE: Czech
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 272944	B1	19910212	CS 1989-1456	19890308

OTHER SOURCE(S): CASREACT 117:212134; MARPAT 117:212134
GI



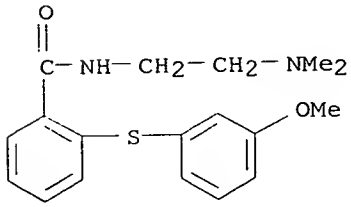
I

AB The title compds. (I; R1 = R2 = H, Et, Pr, Me2CH; R1 = Me2NCH2CH2, R2 = H, Me) [II; R = (OH)n; n = 1, 2] and their salts, were prepd. by demethylation of the parent anisoles II (R = (OMe)n; n as above)] by heating with pyridine-HCl or 48% HBr, or by BBr3 at the ambient temp., followed by neutralization of the resulting bases. Thus, 2-(2-methoxyphenylthio)benzoic acid was converted (93%) to its chloride, then amidated (95%) by aq. NH3, and the amide reduced (77%, isolated as the HCl salt) by LiAlH4 in Et2O. The resulting 2-[(2-methoxyphenyl)thio]benzylamine (5.8 g) was stirred and heated at 210-215.degree. with 14 g pyridine-HCl to give 4.1 g title compd. I (R = 2-HO, R1 = R2 = H) (III). The latter had IC50 = 50 mg/L against Pseudomonas aeruginosa, Proteus vulgaris, and Trichophyton mentagrophytes. Approx. 17 I were prepd. and several I (tested as HCl- or maleate salts) had IC50 of 16-128 mg/L in growth inhibition tests with 7 microorganisms. I in mice had oral acute toxicity LD50 of 146-704 mg/kg.

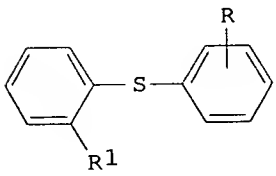
IT **127906-90-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)

CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[(3-methoxyphenyl)thio]- (9CI) (CA INDEX NAME)

(prepn. and reaction of, in prepn. of antimicrobial agent)
RN 127906-90-5 CAPLUS
CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[(3-methoxyphenyl)thio]- (9CI) (CA INDEX NAME)



L55 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1990:458596 CAPLUS
DOCUMENT NUMBER: 113:58596
TITLE: Potential antidepressants: 2-(methoxy- and hydroxyphenylthio)benzylamines as selective inhibitors of 5-hydroxytryptamine re-uptake in the brain
AUTHOR(S): Jilek, Jiri; Sindelar, Karel; Pomykacek, Josef; Kmonicek, Vojtech; Sedivy, Zdenek; Hrubantova, Marta; Holubek, Jiri; Svatek, Emil; Ryska, Miroslav; et al.
CORPORATE SOURCE: Res. Inst. Pharm. Biochem., Prague, 130 60, Czech.
SOURCE: Collect. Czech. Chem. Commun. (1989), 54(12), 3294-338
CODEN: CCCCAK; ISSN: 0010-0765
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 113:58596
GI

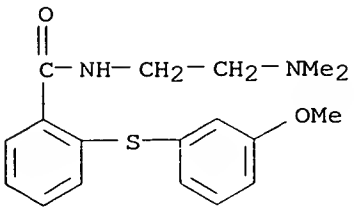


I

AB 2-, 3-, And 4-methoxythiophenol, and 2,4-, 2,5- and 3,4-dimethoxythiophenol were transformed in two steps to (phenylthio)benzoyl chlorides I (R = 2-, 3-, 4-OMe, 2,4-, 2,5-, 3,4-(OMe)2, R1 = COCl), which were reacted with NH3, MeNH2, Me2NH, Et2NH, Pr2NH, and (Me2CH)2NH to give the amides I [R1 = CONH2, CONHMe, CONMe2CONEt2, CONPr2, CON(CHMe2)2]. These were reduced mostly with LiAlH4 to the amines I (R1 = CH2NH2, CH2NMe2 etc.). These methoxylated amines were demethylated either by heating with pyridine hydrochloride or by treatment with BBr3. Some of the -(methoxy- and hydroxyphenylthio)benzylamines prepd., indicated properties of potential antidepressants being highly active and selective inhibitors of 5-hydroxytryptamine re-uptake in the brain structures and having the typical antireserpine activity. The most interesting compd. of the series is I (R = 3-OH, R1 = CH2NMe2) which is undergoing preclin. studies.

IT **127906-90-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and hydride redn. of)
RN 127906-90-5 CAPLUS

CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[(3-methoxyphenyl)thio]- (9CI) (CA INDEX NAME)



L55 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2000 ACS
ACCESSION NUMBER: 1977:552276 CAPLUS
DOCUMENT NUMBER: 87:152276
TITLE: 3-(Heterocyclicthiomethyl)quinoxaline 1,4-dioxides
INVENTOR(S): Urban, Frank J.
PATENT ASSIGNEE(S): Pfizer Inc., USA
SOURCE: U.S., 13 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4038392	A	19770726	US 1975-622057	19751014
NL 7610317	A	19770418	NL 1976-10317	19760916
BE 846532	A1	19770324	BE 1976-1007643	19760924
FR 2327784	A1	19770513	FR 1976-28849	19760924
FR 2327784	B1	19781117		
JP 52048679	A2	19770418	JP 1976-115729	19760927
DE 2645787	A1	19770421	DE 1976-2645787	19761009
PRIORITY APPLN. INFO.:			US 1975-622057	19751014

GI For diagram(s), see printed CA Issue.
AB Quinoxaline dioxides I (R = CO2Me, CONH2, substituted carbamoyl, CH2OH, Ac, H; R1 = CH2SR2, CH2SO2R2, CH2SOR2, CH2SO2CH2R2, CH2SO2(CH2)3R2, R2 = N heterocycle) (>100 compds.) were prepd. Thus I (R = CH2OH, R1 = Me) was brominated and treated wtih 1-methyl-2-imidazolethiol to give I (R = CH2OH, R1 = 1-methyl-2-imidazolylthiomethyl), which had min. inhibitory concns. against Streptococcus pyogenes and Escherichia coli 50 and 100 mg/ml.
IT **63205-98-1P 63206-13-3P 63206-17-7P**
63206-29-1P 63206-32-6P 63219-25-0P
64300-90-9P 64300-93-2P 64300-95-4P
RL: BAC (Biological activity or effector, except adverse); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)
(prepn. and bactericidal activity of)
RN 63205-98-1 CAPLUS
CN 2-Quinoxalinecarboxamide, N-[2-(dimethylamino)ethyl]-3-[[(2-pyridinylmethyl)sulfonyl]methyl]-, 1,4-dioxide (9CI) (CA INDEX NAME)

This file supports REGIstRY for direct browsing and searching of all substance data from the REGISTRY file. Enter HELP FIRST for more information.

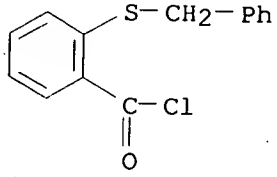
L25 STR
L28 29 SEA FILE=REGISTRY SSS FUL L25
L32 7 SEA FILE=CAOLD ABB=ON L28

=> d ial hitstr 132 1-7; fil hom

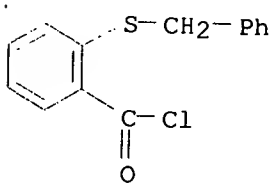
L32 ANSWER 1 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA59:10010f CAOLD
TITLE: 11-(3-dimethylaminopropylidene)-6,11-dihydrodibenz(b,e)thiepin
AUTHOR NAME: Protiva, Miroslav; Rajsner, M.; Votava, Z.; Metysova, J.
DOCUMENT TYPE: Patent

PATENT NO.	KIND	DATE
PI CZ 105590		

INDEX TERM: 1531-77-7 **1531-81-3** 1531-85-7 96175-10-9
IT **1531-81-3**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L32 ANSWER 2 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA59:2772g CAOLD
TITLE: synthetic ataractics - (VII) 11-(3-dimethylaminopropylidene)-6,11-dihydrodibenzo[b,e]thiepins
AUTHOR NAME: Rajsner, Miroslav; Protiva, M.
INDEX TERM: 113-53-1 897-15-4 1531-77-7 **1531-81-3** 1531-85-7 1699-03-2 1699-04-3 1745-46-6 33301-21-2 34129-26-5 96175-10-9
IT **1531-81-3**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L32 ANSWER 3 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA58:4574c CAOLD
TITLE: synthetic medicinals - (VIII) tricyclic thiazepine and thiepin derivs.
AUTHOR NAME: Gadient, Fulvio; Jucker, E.; Lindenmann, A.; Taeschler, M.

SO Eur. Pat. Appl., 276 pp.
CODEN: EPXXDW
DT Patent
LA English
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI EP 301784	A1	19890201	EP 1988-306806	19880725
R: ES, GR				
US 4906282	A	19900306	US 1988-204556	19880615
WO 8900991	A1	19890209	WO 1988-US2459	19880725
W: AU, JP				
RW: AT, BE, CH, DE, FR, GB, IT, LU, NL, SE				
AU 8821334	A1	19890301	AU 1988-21334	19880725
AU 611191	B2	19910606		
EP 386001	A1	19900912	EP 1988-906577	19880725
R: AT, BE, CH, DE, FR, GB, IT, LI, LU, NL, SE				
JP 02504275	T2	19901206	JP 1988-506452	19880725
US 4995901	A	19910226	US 1990-461581	19900105
PRAI US 1987-78191		19870727		
US 1988-204556		19880615		
WO 1988-US2459		19880725		

OS MARPAT 110:207841
AB The sulfonamides JSO2NHC(:W)NRA (I) [J = (un)substituted Ph, naphthyl, thienyl, pyridinyl, pyrazolyl, etc.; W = O, S; R = H, Me; A = (un)substituted 1,2,4-triazolyl, pyrimidinyl, 1,3,5-triazinyl, etc.] are prepd. as herbicides. 2-[Cyano(methoxyimino)methyl]benzenesulfonamide (prepn. given) was reacted with Ph (4,6-dimethoxy-1,3,5-triazin-2-yl)carbamate, in dry acetonitrile, in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene, to give I [J = 2-[MeON:C(CN)]C6H4, W = O, R = H, A = 4,6-dimethoxy-1,3,5-triazin-2-yl] (II). Pre-emergence application of 0.05 kg II/ha controlled velvet-leaf (Abutilon theophrasti), morning-glory (Ipomoea) and other weeds. A wettable powder comprised I [J = 2-[MeON:C(CN)]C6H4, W = O, R = H, A = 4-methoxy-6-methyl-2-pyrimidinyl] 65, dodecylphenol polyethylene glycol ether 2, Na lignin sulfonate 4, Na silicoaluminate 6 and montmorillonite 23%.

L5 ANSWER 8 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1963:454908 CAPLUS
DN 59:54908
OREF 59:10010e-h,10011a
TI 11-(3-Dimethylaminopropylidene)-6,11-dihydrodibenz[b,e]-thiepin
IN Protiva, Miroslav; Rajsner, Miroslav; Votava, Zdenek; Metysova, Jirina
SO ~~4 pp.~~
DT Patent
LA Unavailable

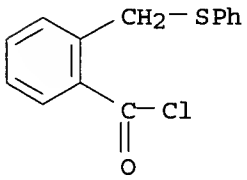
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI CS 105590		19621115	CS	19610608

AB The title compd. (I) has thymoleptic, tranquilizing, antispasmodic, and antihistamine activity. S-Benzylthiosalicylic acid (II) (12.2 g.) in 70 ml. Et2O and 4 g. anhyd. C5H5N treated with 6 g. SOCl2 under cooling, the mixt. kept 2 hrs. at room temp., filtered, and the solid crystd. from C6H6-petr. ether gave the acid chloride (III), m. 118-19.degree.. II (40 g.), 110 g. P2O5 and 750 ml. anhyd. C6H6 refluxed 2 hrs., the mixt. kept overnight at room temp., decompd. by pouring into ice, the C6H6 layer sepd., dried (Na2SO4), and evapd. gave 6.7 g. acid anhydride (IV), m. 106-7.degree. (C6H6-petr. ether). Crude III (prepd. from 12.2 g. II) in 30 ml. PhNO2 treated under cooling and stirring with 12 g. AlCl3 in 30 ml. PhNO2, the mixt. kept 18 hrs. at room temp., poured into a mixt. of ice and dil. HCl, the org. layer sepd., washed (NaOH), dried (K2CO3), evapd. in vacuo, and distd. gave V, b0.1 162-5.degree., m. 85-6.degree. (Et2O-petr. ether). AlCl3 (50 g.) in 70 ml. PhNO2 treated with 41 g. IV

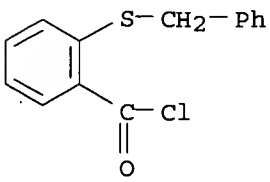
in 130 ml. PhNO2 under cooling and stirring, the mixt. kept 20 hrs. at room temp., decompd. with ice and HCl, the org. layer sepd., washed, dried, and distd, gave V, b1 175-80.degree.. Me2N(CH2)3 MgCl [prepd. from 1.5 g. Mg, several drops of EtBr, and 9 ml. Me2N(CH2)3Cl in 30 ml. anhyd. Et2O] treated with 6.5 g. V in 25 ml. C6H6 under stirring, the mixt. refluxed 18 hrs., cooled, decompd. with 100 ml. 10% NH4Cl, dild. with 100 ml. CHCl3, the org. layer sepd., dried (K2CO3), and evapd. gave 9.0 g. 11-(3-dimethylaminopropyl)-6,11-dihydrodibenz[b,e]thiepin-11-ol (VI), m. 130-1.degree. (C6H6-petr. ether). VI (8.0 g.) and 70 ml. 3N H2SO4 refluxed 5 min., the soln. filtered with C, made alk. with 20% NaOH, extd. with CHCl3, the ext. dried (K2CO3), evapd., and the residue distd. gave 4.3 g. I, b0.2 162-4.degree.; HCl salt m. 215-17.degree. (EtOH-Et2O).

L5 ANSWER 9 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1963:415510 CAPLUS
DN 59:15510
OREF 59:2772g-h,2773a-f
TI Synthetic ataractics. VII. 11-(3-Dimethylaminopropylidene)6,11-dihydrodibenzo[b,e]thiepin
AU Rajsner, M.; Protiva, M.
CS Pharm. Res. Inst., Prague
SO Cesk. Farm. 11 (1962) 404-9
DT Journal
LA Unavailable
AB cf. CA 57, 9817e; 58, 7853g. S-Benzylthiosalicylic acid (I) (40 g.), m. 189.degree., 110 g. P2O5, and 750 ml. anhyd. C6H6 refluxed 2 hrs., the mixt. kept overnight at room temp., decompd. by pouring onto ice, the org. layer sepd., the aq. layer extd. with C6H6, and the org. solns. combined, dried (Na2SO4), and evapd. to dryness gave 29 g. S-benzylthiosalicylic acid anhydride (II), m. 107-7.5.degree. (C6H6-petr. ether). Hydrolysis of II with boiling NaOH in aq. EtOH gave I. I (10 g.) and 25 ml. SOCl2 refluxed till the evolution of gaseous products ceased, the mixt. evapd. in vacuo to dryness, and the residue mixed with EtOH gave 5.8 g. bis(thiosalicylic acid) dichloride, m. 159-61.degree. (CHCl3-petr. ether). I (12.2 g.) in 70 ml. Et2O treated with 4 ml. anhyd. C5H5N and then treated under cooling and shaking with 6 g. SOCl2, the mixt. kept 2 hrs. at room temp. and dild. with petr. ether, the solid filtered off, and the filtrate extd. with 200 ml. C6H6, the ext. filtered, and the soln. evapd. in vacuo to dryness gave 6.5 g. S-benzylthiosalicylic acid chloride (III), m. 117-19.degree. (C6H6-petr. ether), v (Nujol) 710, 750-80, 1260-75, 1465, 1495-1570-90, 1680 cm.-1 EtONa (prepd. from 92 g. Na and 1400 ml. anhyd. EtOH) treated with 440.3 g. PhSH and 536.5 g. phthalide, the mixt. refluxed 4.5 hrs., the greater part of EtOH distd. in vacuo, the residue dissolved in 3 l. H2O, the soln. filtered, the filtrate cooled, and acidified with HCl gave 920 g. o-(phenylthiomethyl)- benzoic acid (IV), m. 113-16.degree. (80% EtOH). IV (24.4 g.) and 50 ml. SOCl2 kept 20 min. at room temp., the mixt. heated to 60.degree. till evolution of gaseous products ceased and evapd. in vacuo, and the residue distd. gave 17 g. acid chloride of IV, b0.5 142-50.degree.. AlCl3 (50 g.) in 70 ml. PhNO2 cooled with ice, treated dropwise with stirring with 41 g. II in 130 ml. PhNO2, the mixt. kept 20 hrs. at room temp., poured onto ice and dil. HCl, the org. layer sepd., washed (dil. HCl, dil. NaOH), dried (K2CO3), evapd. in vacuo to dryness, and the residue distd. gave 5.3 g. 6,11-dihydrodibenzo[b,e]thiepin-11-one (V), b1 175-80.degree., m. 80-7.degree. (Et2O-petr. ether), v (CCl4, Nujol) 703, 733, 759, 777, 800, 930, 1045, 1072, 1118, 1152, 1249, 1291-1300, 1428, 1452, 1463, 1595, 1652 cm.-1 III (6.5 g.) in 30 ml. PhNO2 treated under external cooling dropwise with 12 g. AlCl3 in 30 ml. PhNO2, the mixt. kept 18 hrs. at room temp., and worked up gave 1.4 g. V, b0.1 162-5.degree., m. 86-7.degree.. IV (160 g.) cyclized 1 hr. with polyphosphoric acid (prepd. from 510 g. P2O5 and 340 ml. 90% H3PO4) at 90.degree., the mixt. poured onto 2 kg. ice and H2O and extd. with C6H6, and the org. layer washed (H2O, 5% NaOH), dried (K2CO3), and evapd. gave 113.5 g. V, m. 86-7.degree. (EtOH). V (2.3 g.) in 30 ml. anhyd. MeOH reduced with 0.6 g. NaBH4, the mixt. refluxed 10

```
=> s e204
L1            1 "BENZOYL CHLORIDE, 2-((PHENYLTHIO)METHYL)-"/CN

=> s e201
L2            1 "BENZOYL CHLORIDE, 2-((PHENYLMETHYL)THIO)-"/CN

=> d l1

L1   ANSWER 1 OF 1  REGISTRY  COPYRIGHT 2003 ACS
RN   1699-04-3  REGISTRY
CN   Benzoyl chloride, 2-[(phenylthio)methyl]- (9CI)  (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN   o-Toluoyl chloride, .alpha.-(phenylthio)- (7CI, 8CI)
FS   3D CONCORD
MF   C14 H11 Cl O S
LC   STN Files:   BEILSTEIN*, CA, CAOLD, CAPLUS, CASREACT
        (*File contains numerically searchable property data)
```




```
**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

              9 REFERENCES IN FILE CA (1962 TO DATE)
              9 REFERENCES IN FILE CAPLUS (1962 TO DATE)
              6 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

=> d l2

L2   ANSWER 1 OF 1  REGISTRY  COPYRIGHT 2003 ACS
RN   1531-81-3  REGISTRY
CN   Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI)  (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN   Benzoyl chloride, o-(benzylthio)- (6CI, 7CI, 8CI)
OTHER NAMES:
CN   o-(Benzylthio)benzoyl chloride
CN   S-Benzylthiosalicylic acid chloride
FS   3D CONCORD
MF   C14 H11 Cl O S
LC   STN Files:   BEILSTEIN*, CA, CAOLD, CAPLUS, CASREACT, TOXCENTER, USPATFULL
        (*File contains numerically searchable property data)
```




```
**PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT**

              13 REFERENCES IN FILE CA (1962 TO DATE)
              13 REFERENCES IN FILE CAPLUS (1962 TO DATE)
```

5 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

=> FIL REGISTRY

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	13.80	14.01

FILE 'REGISTRY' ENTERED AT 18:24:37 ON 23 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2003 American Chemical Society (ACS)

Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 22 APR 2003 HIGHEST RN 503805-80-9
DICTIONARY FILE UPDATES: 22 APR 2003 HIGHEST RN 503805-80-9

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2003

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. See HELP PROPERTIES for more information. See STNote 27, Searching Properties in the CAS Registry File, for complete details:
http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

=> SET TERMSET E#

SET COMMAND COMPLETED

=> DEL SEL Y

=> SEL L1 1 RN

E1 THROUGH E1 ASSIGNED

=> S E1/RN

L3 1 1699-04-3/RN

=> SET TERMSET LOGIN

SET COMMAND COMPLETED

=> FIL CAPLUS

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.48	14.49

FILE 'CAPLUS' ENTERED AT 18:24:42 ON 23 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)

held by the publishers listed in the PUBLISHER (PB) field (available for records published or updated in Chemical Abstracts after December 26, 1996), unless otherwise indicated in the original publications. The CA Lexicon is the copyrighted intellectual property of the American Chemical Society and is provided to assist you in searching databases on STN. Any dissemination, distribution, copying, or storing of this information, without the prior written consent of CAS, is strictly prohibited.

FILE COVERS 1907 - 23 Apr 2003 VOL 138 ISS 17
FILE LAST UPDATED: 22 Apr 2003 (20030422/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> S L3

L4 9 L3

=> s l2
L5 13 L2

=> d l5 1-13 bib,ab

L5 ANSWER 1 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 2002:814891 CAPLUS
DN 137:325335
TI Preparation of (hetero)arylamides as inhibitors of microsomal triglyceride transfer protein
IN Booth, Richard John; Lee, Helen Tsenwhei; Pontrello, Jason Keith; Ramharack, Randy Ranjee; Roth, Bruce David
PA USA
SO U.S. Pat. Appl. Publ., 27 pp., Cont.-in-part of U.S. Ser. No. 422,568.
CODEN: USXXCO
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2002156281	A1	20021024	US 2001-21633	20011212
PRAI	US 1998-107119P	P	19981105		
	US 1999-422568	B2	19991021		

OS MARPAT 137:325335
AB R3(CH2)nNR1COR2 [I, R1 = (substituted) pyridyl, pyridylmethyl, Ph, quinolyl, benzothienyl, etc.; R2 = Ph, PhCH2OC6H4, PhCH2SC6H4, PhCH2SOC6H4, naphthylmethyl, benzodioxanyl, benzothienyl, amino, aminoalkyl, etc.; R3 = biphenyl, benzothienyl, tetramethyltetralinyl, naphthalenyl; n = 0-2], were prepd. Thus, reaction of 2-ethoxy-N-pyridin-3-ylbenzamide and 2-phenylbenzyl bromide gave N-biphenyl-2-ylmethyl-2-ethoxy-N-pyridin-3-ylbenzamide. The latter inhibited lipoprotein A3 prodn. with IC50 = 0.9 .mu.M. The present invention also provides pharmaceutical compns. comprising I and methods of treatment of atherosclerosis, obesity, restenosis, coronary heart disease, hyperlipoproteinemia, hypercholesterolemia, and hypertriglyceridemia.

L5 ANSWER 2 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 2001:166492 CAPLUS
DN 134:326427
TI A novel synthesis of [1]benzothieno[3,2-b][1]benzofuran
AU Cernovska, Katerina; Nic, Miloslav; Pihera, Pavel; Svoboda, Jiri
CS Department of Organic Chemistry, Institute of Chemical Technology, Prague, Prague, 16628/6, Czech Rep.
SO Collection of Czechoslovak Chemical Communications (2000), 65(12),

1939-1949
CODEN: CCCCAK; ISSN: 0010-0765
PB Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic
DT Journal
LA English
OS CASREACT 134:326427
AB A new synthesis of the title compd. based on the formation of the furan ring in the key step was elaborated. Me 2-methoxy[1]benzothieno[3,2-b][1]benzofuran-7-carboxylate was prepd. by this methodol. as a new type of a core for liq. crystal synthesis.
RE.CNT 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 3 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1993:517742 CAPLUS
DN 119:117742
TI Organic nitrates, methods for preparing same, and use thereof for treating cardiovascular diseases
IN Nallet, Jean Pierre; Dreux, Jacques; Berdeaux, Alain; Richard, Vincent; Martorana, Piero; Bohn, Helmut
PA Laboratoires Hoechst, Fr.
SO PCT Int. Appl., 96 pp.
CODEN: PIXXD2
DT Patent
LA French
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	WO 9303037	A1	19930218	WO 1992-EP1746	19920801
	W: CA, HU, JP, KR, US				
	RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE				
	FR 2680173	A1	19930212	FR 1991-10039	19910807
	FR 2680173	B1	19950505		
	CA 2113922	AA	19930218	CA 1992-2113922	19920801
	EP 530887	A1	19930310	EP 1992-202500	19920801
	R: PT				
	EP 604459	A1	19940706	EP 1992-917213	19920801
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, SE				
	JP 07500817	T2	19950126	JP 1992-503265	19920801
	HU 70546	A2	19951030	HU 1994-327	19920801
	US 5591758	A	19970107	US 1993-971812	19930504
PRAI	FR 1991-10039		19910807		
	WO 1992-EP1746		19920801		

OS MARPAT 119:117742
AB Org. nitrates RCOAnYB [I; R = many possible groups, particularly S-contg. residues, including thiazolidines and S-contg. amino acids; A = particularly CH2 or a substituted amino acid; n = 0, 1, >1; Y = O, NH; B = particularly dianhydro-1,4:3,6-hexitol mononitrate residues, itol nitrate residues, inositol nitrate residues] were prepd. as vasorelaxants for treatment of cardiovascular diseases, particularly angina pectoris, and show diminished tachyphylaxis. For example, amidation of 1,4:3,6-dianhydro-5-deoxy-5-amino-L-iditol 2-nitrate with N-(tert-butoxycarbonyl)glycine (72%), followed by deprotection with HCl-MeOH (85%), neutralization of the HCl salt (90%), a 2nd amidation with N-(tert-butoxycarbonyl)-L-thioproline using DCC (71%), and deprotection with HCl-EtOAc (76%), gave title compd. L-II as the HCl salt (III). Prepns. of over 55 I and 17 precursors, and detailed results of a variety of hemodynamic tests on several I are given. In comparison with isosorbide mononitrate, III showed higher potency, longer duration of action, and an absence of tachyphylaxis.

L5 ANSWER 4 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1991:655826 CAPLUS

DN 115:255826
TI Preparation of propanediamine derivatives as ligands for radioactive isotopes, their metal complexes, and their use in diagnosis and therapy
IN Neumeier, Reinhard; Kramp, Wolfgang; Maecke, Helmut R.
PA Institut fuer Diagnostikforschung G.m.b.H., Germany
SO Eur. Pat. Appl., 29 pp.
CODEN: EPXXDW
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 417870	A2	19910320	EP 1990-250214	19900820
	EP 417870	A3	19910626		
	EP 417870	B1	19940720		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE				
	DE 3930674	A1	19910321	DE 1989-3930674	19890911
	NO 9003551	A	19910312	NO 1990-3551	19900813
	NO 173234	B	19930809		
	NO 173234	C	19931117		
	HU 59370	A2	19920528	HU 1990-5026	19900815
	CA 2023595	AA	19910312	CA 1990-2023595	19900820
	ES 2060002	T3	19941116	ES 1990-250214	19900820
	ZA 9006634	A	19910626	ZA 1990-6634	19900821
	US 5302370	A	19940412	US 1990-572140	19900822
	AU 9061290	A1	19910314	AU 1990-61290	19900823
	AU 641421	B2	19930923		
	IL 95547	A1	19960514	IL 1990-95547	19900831
	DD 297636	A5	19920116	DD 1990-343845	19900905
	JP 03188048	A2	19910816	JP 1990-239148	19900911
PRAI	DE 1989-3930674		19890911		

OS MARPAT 115:255826
AB The title ligands [I; R1, R2, R5 = H, (HO-substituted) C1-6 alkyl; R3, R4 = H, (amino)C1-6 alkyl, HO2CCH2, (C1-6 alkoxycarbonyl)methyl or -benzyl; R6 = C1-6 alkylene; R7, R8 = H, C1-6 alkyl; B, B1 = Ph, 2-HSC6H4, naphthyl, thienyl, pyrrolyl, all optionally substituted by 1-3 HO), CH(NO)R9; R9 = C1-6 alkyl; R1R9, R2R9 can form a 5- or 6-membered ring with (CH2)3 or (CH2)4; A = functional group Z, a compd. T bound to R6 via Z and capable of accumulating itself in lesions or specific tissues, e.g. an enzyme, amino acid, saccharide, a growth factor, esp. a monoclonal antibody or its fragments, biotin, and misonidazole; Z = amino, carboxy, HO, oxiranyl, aminophenyl, C2-6 alkenyl, etc.], useful in tumor diagnosis and therapy, were prepd. Condensation of 4-O2NC6H4CH(CH2NH2)2 [prepn. from CH2(CO2Et)2 and 4-O2NC6H4CH2Br given] with 2-chloro-2-methyl-3-nitrosobutane gave 27% 6-(4'-nitrobenzyl)-3,3,9,9-tetramethyl-4,8-diazaundecane-2,10-dione dioxime. This was reduced (26%) to its 4'-aminobenzyl analog, chelated by Cu(OAc)2 (45%), the Cu-chelate coupled (75%) at position 4' with biotin N-hydroxysuccinimide ester, the resulting biotin conjugate decomplexed (41%) by KCN, and the ligand recomplexed with a radioactive tracer: technetium-99m (200 .mu.Ci). A rat left hind leg muscle was injected with 20 .mu.L of a com. streptavidin-Sepharose conjugate and, 30 min later, with 5 .mu.g (i.v.) of the latter chelate (purity >90%). After 4 h, the radioactivity in the left hind leg was 14-fold higher than in the right hind leg, and it contained 1.4% of the total of the applied dosis/g muscle.

L5 ANSWER 5 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1989:646719 CAPLUS
DN 111:246719
TI Molybdenum(VI)-dioxo complexes with linear and tripodal tetradentate ligands: models for the molybdenum(VI/V) centers of the molybdenum hydroxylases and related enzymes. 1. Syntheses and structures
AU Hinshaw, Carol J.; Peng, Gang; Singh, Raghuvir; Spence, Jack T.; Enemark, John H.; Bruck, Michael; Kristofzski, John; Merbs, Shannath L.; Ortega,

Richard B.; Wexler, Pamela A.
CS Dep. Chem. Biochem., Utah State Univ., Logan, UT, 84322-0300, USA
SO Inorganic Chemistry (1989), 28(25), 4483-91
CODEN: INOCAJ; ISSN: 0020-1669
DT Journal
LA English
AB As models for the molybdenum(VI/V) centers of the molybdenum hydroxylases and related enzymes, 15 new Mo(VI)-dioxo complexes (MoO2L) with tetradentate ligands were prepd. and characterized. The effects of coordinating groups (N2S2, N2OS, and N2O2), chelate ring size (five and six members), ligand geometry (linear and tripodal), and steric bulk were studied. X-ray crystal structures were obtained for seven of the complexes. While minor differences, attributed to these features, are evident, the structures have remarkably similar Mo-ligand bond lengths and bond angles and all have distorted-octahedral geometry. The oxo groups are cis to one another and to the thiolate or phenolate groups of the ligands. The N atoms are approx. trans to the oxo groups, and the Mo-N bonds are relatively long (>2.34 .ANG.), with the bond length correlated with the size of the trans O=Mo-N bond angle. The Mo=O and M-S(thiolate) bond lengths are comparable to those detd. by EXAFS spectroscopy for the Mo centers of the enzymes. The relevance of the results to the structures of the Mo centers of the enzymes is discussed.

L5 ANSWER 6 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1989:515023 CAPLUS
DN 111:115023
TI Pyrrole derivatives as cardiotonics, process for their preparation and pharmaceutical compositions containing them
IN Dixon, John; Baxter, Andrew John Gilby; Manners, Carol Nancy; Teague, Simon
PA Fisons PLC, UK
SO Eur. Pat. Appl., 69 pp.
CODEN: EPXXDW
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 300688	A1	19890125	EP 1988-306464	19880714
	R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, LU, NL, SE				
	DK 8804049	A	19890122	DK 1988-4049	19880720
	JP 01061455	A2	19890308	JP 1988-179286	19880720
PRAI	GB 1987-17193		19870721		
	GB 1987-30116		19871224		

OS MARPAT 111:115023
AB Title compds. I [R1 = R11, NHR11, NHCO2R11 wherein R11 = H, C1-6 alkyl; R2, R5 = OH, halo, NO2, etc.; G = (CH2)zWy in which W = CO, SOq, etc.; q = 0-2; z = 0-3; y = 0 or 1 (or 2 provided W = CO); up to 2 of the methylene segments in the chain (CH2)z are optionally replaced by NH and one segment is optionally replaced by O, etc.; the chain is optionally unsatd. and optionally substituted by C1-6 alkyl, alkoxy, etc.; A = (substituted) 5- or 6-membered ring or a bicyclic or tricyclic fused ring system; R3 = H, NO2, CN, halo, etc.; several provisos are given], useful as cardiotonics (no data), were prepd. A mixt. of 2-((4-nitrophenyl)thio)benzoyl chloride, Me 2,5-dimethyl-1H-pyrrole-3-carboxylate, and AlCl3 in CH2Cl2 was stirred at room temp. for 16 h to give Me 2,5-dimethyl-4-(2-((4-nitrophenyl)thio)benzoyl)-1H-pyrrole-3-carboxylate.

L5 ANSWER 7 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1989:207841 CAPLUS
DN 110:207841
TI Herbicidal sulfonamides
IN Rorer, Morris Padgett
PA du Pont de Nemours, E. I., and Co., USA

SO Eur. Pat. Appl., 276 pp.
CODEN: EPXXDW
DT Patent
LA English
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 301784	A1	19890201	EP 1988-306806	19880725
	R: ES, GR				
	US 4906282	A	19900306	US 1988-204556	19880615
	WO 8900991	A1	19890209	WO 1988-US2459	19880725
	W: AU, JP				
	RW: AT, BE, CH, DE, FR, GB, IT, LU, NL, SE				
	AU 8821334	A1	19890301	AU 1988-21334	19880725
	AU 611191	B2	19910606		
	EP 386001	A1	19900912	EP 1988-906577	19880725
	R: AT, BE, CH, DE, FR, GB, IT, LI, LU, NL, SE				
	JP 02504275	T2	19901206	JP 1988-506452	19880725
	US 4995901	A	19910226	US 1990-461581	19900105
PRAI	US 1987-78191		19870727		
	US 1988-204556		19880615		
	WO 1988-US2459		19880725		

OS MARPAT 110:207841
AB The sulfonamides JSO2NHC(:W)NRA (I) [J = (un)substituted Ph, naphthyl, thienyl, pyridinyl, pyrazolyl, etc.; W = O, S; R = H, Me; A = (un)substituted 1,2,4-triazolyl, pyrimidinyl, 1,3,5-triazinyl, etc.] are prepd. as herbicides. 2-[Cyano(methoxyimino)methyl]benzenesulfonamide (prepn. given) was reacted with Ph (4,6-dimethoxy-1,3,5-triazin-2-yl)carbamate, in dry acetonitrile, in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene, to give I [J = 2-[MeON:C(CN)]C6H4, W = O, R = H, A = 4,6-dimethoxy-1,3,5-triazin-2-yl] (II). Pre-emergence application of 0.05 kg II/ha controlled velvet-leaf (Abutilon theophrasti), morning-glory (Ipomoea) and other weeds. A wettable powder comprised I [J = 2-[MeON:C(CN)]C6H4, W = O, R = H, A = 4-methoxy-6-methyl-2-pyrimidinyl] 65, dodecylphenol polyethylene glycol ether 2, Na lignin sulfonate 4, Na silicoaluminate 6 and montmorillonite 23%.

L5 ANSWER 8 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1963:454908 CAPLUS
DN 59:54908
OREF 59:10010e-h,10011a
TI 11-(3-Dimethylaminopropylidene)-6,11-dihydrodibenz[b,e]-thiepin
IN Protiva, Miroslav; Rajsner, Miroslav; Votava, Zdenek; Metysova, Jirina
SO ~~4 pp.~~
DT Patent
LA Unavailable

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	CS 105590		19621115	CS	19610608

AB The title compd. (I) has thymoleptic, tranquilizing, antispasmodic, and antihistamine activity. S-Benzylthiosalicylic acid (II) (12.2 g.) in 70 ml. Et2O and 4 g. anhyd. C5H5N treated with 6 g. SOCl2 under cooling, the mixt. kept 2 hrs. at room temp., filtered, and the solid crystd. from C6H6-petr. ether gave the acid chloride (III), m. 118-19.degree.. II (40 g.), 110 g. P2O5 and 750 ml. anhyd. C6H6 refluxed 2 hrs., the mixt. kept overnight at room temp., decompd. by pouring into ice, the C6H6 layer sepd., dried (Na2SO4), and evapd. gave 6.7 g. acid anhydride (IV), m. 106-7.degree. (C6H6-petr. ether). Crude III (prepd. from 12.2 g. II) in 30 ml. PhNO2 treated under cooling and stirring with 12 g. AlCl3 in 30 ml. PhNO2, the mixt. kept 18 hrs. at room temp., poured into a mixt. of ice and dil. HCl, the org. layer sepd., washed (NaOH), dried (K2CO3), evapd. in vacuo, and distd. gave V, b0.1 162-5.degree., m. 85-6.degree. (Et2O-petr. ether). AlCl3 (50 g.) in 70 ml. PhNO2 treated with 41 g. IV

in 130 ml. PhNO2 under cooling and stirring, the mixt. kept 20 hrs. at room temp., decompd. with ice and HCl, the org. layer sepd., washed, dried, and distd, gave V, b1 175-80.degree.. Me2N(CH2)3 MgCl [prepd. from 1.5 g. Mg, several drops of EtBr, and 9 ml. Me2N(CH2)3Cl in 30 ml. anhyd. Et2O] treated with 6.5 g. V in 25 ml. C6H6 under stirring, the mixt. refluxed 18 hrs., cooled, decompd. with 100 ml. 10% NH4Cl, dild. with 100 ml. CHCl3, the org. layer sepd., dried (K2CO3), and evapd. gave 9.0 g. 11-(3-dimethylaminopropyl)-6,11-dihydrodibenz[b,e]thiepin-11-ol (VI), m. 130-1.degree. (C6H6-petr. ether). VI (8.0 g.) and 70 ml. 3N H2SO4 refluxed 5 min., the soln. filtered with C, made alk. with 20% NaOH, extd. with CHCl3, the ext. dried (K2CO3), evapd., and the residue distd. gave 4.3 g. I, b0.2 162-4.degree.; HCl salt m. 215-17.degree. (EtOH-Et2O).

L5 ANSWER 9 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1963:415510 CAPLUS
DN 59:15510
OREF 59:2772g-h,2773a-f
TI Synthetic ataractics. VII. 11-(3-Dimethylaminopropylidene)6,11-dihydrodibenzo[b,e]thiepin
AU Rajsner, M.; Protiva, M.
CS Pharm. Res. Inst., Prague
SO Cesk. Farm. 11 (1962) 404-9
DT Journal
LA Unavailable
AB cf. CA 57, 9817e; 58, 7853g. S-Benzylthiosalicylic acid (I) (40 g.), m. 189.degree., 110 g. P2O5, and 750 ml. anhyd. C6H6 refluxed 2 hrs., the mixt. kept overnight at room temp., decompd. by pouring onto ice, the org. layer sepd., the aq. layer extd. with C6H6, and the org. solns. combined, dried (Na2SO4), and evapd. to dryness gave 29 g. S-benzylthiosalicylic acid anhydride (II), m. 107-7.5.degree. (C6H6-petr. ether). Hydrolysis of II with boiling NaOH in aq. EtOH gave I. I (10 g.) and 25 ml. SOCl2 refluxed till the evolution of gaseous products ceased, the mixt. evapd. in vacuo to dryness, and the residue mixed with EtOH gave 5.8 g. bis(thiosalicylic acid) dichloride, m. 159-61.degree. (CHCl3-petr. ether). I (12.2 g.) in 70 ml. Et2O treated with 4 ml. anhyd. C5H5N and then treated under cooling and shaking with 6 g. SOCl2, the mixt. kept 2 hrs. at room temp. and dild. with petr. ether, the solid filtered off, and the filtrate extd. with 200 ml. C6H6, the ext. filtered, and the soln. evapd. in vacuo to dryness gave 6.5 g. S-benzylthiosalicylic acid chloride (III), m. 117-19.degree. (C6H6-petr. ether), v (Nujol) 710, 750-80, 1260-75, 1465, 1495-1570-90, 1680 cm.-1 EtONa (prepd. from 92 g. Na and 1400 ml. anhyd. EtOH) treated with 440.3 g. PhSH and 536.5 g. phthalide, the mixt. refluxed 4.5 hrs., the greater part of EtOH distd. in vacuo, the residue dissolved in 3 l. H2O, the soln. filtered, the filtrate cooled, and acidified with HCl gave 920 g. o-(phenylthiomethyl)- benzoic acid (IV), m. 113-16.degree. (80% EtOH). IV (24.4 g.) and 50 ml. SOCl2 kept 20 min. at room temp., the mixt. heated to 60.degree. till evolution of gaseous products ceased and evapd. in vacuo, and the residue distd. gave 17 g. acid chloride of IV, b0.5 142-50.degree.. AlCl3.(50 g.) in 70 ml. PhNO2 cooled with ice, treated dropwise with stirring with 41 g. II in 130 ml. PhNO2, the mixt. kept 20 hrs. at room temp., poured onto ice and dil. HCl, the org. layer sepd., washed (dil. HCl, dil. NaOH), dried (K2CO3), evapd. in vacuo to dryness, and the residue distd. gave 5.3 g. 6,11-dihydrodibenzo[b,e]thiepin-11-one (V), b1 175-80.degree., m. 80-7.degree. (Et2O-petr. ether), v (CCl4, Nujol) 703, 733, 759, 777, 800, 930, 1045, 1072, 1118, 1152, 1249, 1291-1300, 1428, 1452, 1463, 1595, 1652 cm.-1 III (6.5 g.) in 30 ml. PhNO2 treated under external cooling dropwise with 12 g. AlCl3 in 30 ml. PhNO2, the mixt. kept 18 hrs. at room temp., and worked up gave 1.4 g. V, b0.1 162-5.degree., m. 86-7.degree.. IV (160 g.) cyclized 1 hr. with polyphosphoric acid (prepd. from 510 g. P2O5 and 340 ml. 90% H3PO4) at 90.degree., the mixt. poured onto 2 kg. ice and H2O and extd. with C6H6, and the org. layer washed (H2O, 5% NaOH), dried (K2CO3), and evapd. gave 113.5 g. V, m. 86-7.degree. (EtOH). V (2.3 g.) in 30 ml. anhyd. MeOH reduced with 0.6 g. NaBH4, the mixt. refluxed 10

min. and evapd., the residue decompd. with 20 ml. H2O, extd. with CHCl3, and the ext. dried (MgSO4) and evapd. gave 2.1 g. 6,11-dihydrodibenzo[b,e]thiepin-11-ol, m. 107-8.degree. (C6H6-petr. ether). V (2.3 g.) in 15 ml. AcOH treated with 1 ml. 30% H2O2, the mixt. kept 48 hrs. at room temp., and dild. with 70 ml. H2O gave 2.0 g. 6,11-dihydrodibenzo [b,e] thiepin-11-one 5-oxide, m. 97-100.degree. (EtOH). V (2.3 g.) in 15 ml. AcOH- treated with 4.6 ml. 30% H2O2 and the mixt. refluxed 3 hrs. and cooled gave 2.15 g. 6,11-dihydrodibenzo [b,e]thiepin-11-one 5,5dioxide, m. 127-8.degree. (EtOH). Me2N(CH2)3MgCl [from 38.6 g. Mg, 5 ml. EtBr, and 193 g. Me2N(CH2)3Cl in 600 ml. anhyd. Et2O] refluxed and treated dropwise with 185 g. V in 750 ml. C6H6, the mixt. stirred and refluxed 18 hrs., cooled, and decompd. with 1500 ml. 10% NH4Cl, the org. layer sepd., dried (K2CO3), and partially evapd., and the residue treated with 500 ml. petr. ether gave 154 g. 11-(3-dimethylaminopropyl) 6,11-dihydrodibenzo[b,e]thiepin-11-ol (VI), m. 130-1.degree. (C6H6 petr. ether), .lambda. 261 m.mu. (log .epsilon. 4.0) in MeOH, v (CHCl3) 770-90, 1110-70, 1430, 1460, 1590, 2780-2825 cm.-1 VI (130 g.) and 1000 ml. 3N H2SO4 refluxed 20 min., the mixt. cooled, made alk. with 25% NaOH, and extd. with Et2O, the ext. dried (K2CO3) and evapd., and the residue (120.5 g.) dissolved in 100 ml. anhyd. EtOH and acidified with anhyd. HCl in Et2O gave 123 g. HCl salt of VII, m. 218-21.degree. (EtOH-Et2O), .lambda. 232, 260, 309 m.mu. (log .epsilon. 4.41, 3.97, 3.53) in MeOH, v (CHCl3) 760-90, 1430, 1460, 1590, 2350, 3400 cm.-1; the base b0.2 162-4.degree.. The HCl salt of VII (prothiadene) has mild tranquilizing activity and is being clinically tested as an antidepressive drug.

L5 ANSWER 10 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1963:27348 CAPLUS
DN 58:27348
OREF 58:4574c-h,4575a-h,4576a-d
TI Synthetic medicinals. VIII. New-type tricyclic thiazepine and thiepin derivatives
AU Gadient, F.; Jucker, E.; Lindenmann, A.; Taeschler, M.
CS Sandoz A.-G., Basel, Switz.
SO Helv. Chim. Acta (1962), 45, 1800-70
DT Journal
LA German
AB cf. CA 56, 1532i. The syntheses and pharmacol. properties were described of new type tricyclic compds., derivs. of 5,11-dihydrobenzo[b]pyrido[2,3-e]-1,4-thiazepine (I)and of 6,11-dihydrodibenzo[b,e]thiepin (II). To 16.0 g. 3-hydroxymethylpyridine N-oxide in 75 ml. CHCl3 was added dropwise during 30 min. 43.0 g. SOCl2 under H2O cooling, the whole refluxed 2 hrs., and cooled in ice H2O to give 3-chloromethylpyridine N-oxide HCl salt (III.HCl), m. 98-100.degree. (CHCl3). III.HCl (9.0 g.) suspended in 60 ml. CHCl3, shaken with 4.2 g. NaHCO3 in 40 ml. H2O, the aq. phase sepd., extd. twice with 60 ml. CHCl3, the combined CHCl3 solns. dried, and concd. in vacuo until crystn. commenced gave III, m. 135-7.degree. (CHCl3). III (34.0 g.) added during 30 min. to 100 ml. POCl3 at 25-30.degree., the whole refluxed 2 hrs., the excess POCl3 completely removed in vacuo, the residue dissolved in 100 ml. CHCl3, the soln. washed with 100 g. ice H2O, dried, and fractionated gave 2-chloro-3-chloromethylpyridine (IV), b13 115.degree.. IV (8.1 g.) added rapidly dropwise to 6.25 g. 2-H2NC6H4SH and 2.0 g. NaOH in 40 ml. EtOH and 10 ml. H2O in an N atm., the whole refluxed 70 min., cooled, filtered, the filtrate concd. in vacuo, the residue dissolved in 100 ml. CHCl3, the soln. extd. with 2 50-ml. portions 5N HCl, the combined exts. neutralized with 5N NaOH, the product isolated with CHCl3, and distd. gave 2-chloro-3-[(2-aminophenyl)thiomethyl]pyridine (V), b0.02 150-60.degree. (air bath temp.). V (70.0 g.) and 6.0 g. PhNMe2 in 130 ml. xylene refluxed 4 hrs., the resulting ppt. filtered off, partitioned between 200 ml. CHCl3 and 100 ml. 10% aq. NaHCO3, the CHCl3 layer washed neutral with H2O, dried, and concd. deposited I, m. 123-5.degree. (C6H6). I (3.3 g.) and 900 mg. 50% NaH in oil suspension in 60 ml. xylene heated 2 hrs. at 160.degree., the whole treated dropwise

during 1 hr. with 2.5 g. 2-(2-chloroethyl)-1-methyl-piperidine in 10 ml. xylene, kept 3 hrs. at 160.degree. cooled, treated with 3 g. NH4Cl in 30 ml. H2O, filtered through diatomaceous earth, the xylene layer in the filtrate sepd., washed with 50 ml. H2O, extd. with 100 ml. 15% aq. tartaric acid, the ext. washed with 20 ml. C6H6, made alk. with 5N NaOH, and the product isolated with C6H6 gave 11-[2-(1-methyl-2-piperidyl)ethyl] deriv. (VI) of I, oil, which was purified on Al2O3 with C6H6. Purified VI (3.4 g.) in 10 ml. MeOH treated with 3.8 g. (76% moist) 1,5-naphthalenedisulfonic acid in 5 ml. MeOH and 1 ml. H2O and kept at room temp. gave VI 1,5-naphthalenedisulfonate (VII) hydrate, m. 235-50.degree. (decompn.) (aq. MeOH). Similarly were prepd. 11-(3-dimethylaminopropyl) deriv. (VIII) of I 1,5-naphthalenedisulfonate, m. 175-85.degree. (decompn.) (aq. EtOH), and 11-(2-dimethylaminopropyl) deriv. (IX) of I 1,5-naphthalenedisulfonate, m. 170-80.degree. (decompn.) (aq. EtOH). 2-MeC6H4CO2Et (IXa), 107 g. SO2Cl2, and 760 mg. Bz2O2 heated at 60.degree. (oil bath) while irradiating with ultraviolet light, when gas evolution stopped the unchanged IXa distd. in vacuo (at 13 mm.), and the residue fractionated gave 2-ClCH2C6H4CO2Et (X), b0.03 100-2.degree.. X (87.0 g.) added dropwise to 48.2 g. PhSH and 17.5 g. NaOH in 90 ml. H2O and 350 ml. EtOH, the whole refluxed 75 min., cooled, filtered, the filtrate concd. in vacuo, the residue dissolved in 300 ml. CHCl3, the soln. washed with 50 ml. ice cold N NaOH and with H2O until neutral, dried, and fractionated gave 2-(4-RC6H4SCH2)C6H4CO2R' (XI) (R = H, R' = Et), b0.2 140-2.degree.. The following XI (R' = Et) were similarly prepd. (R and b.p./mm. given): Cl, 176-8.degree./0.1: Me, 145-50.degree./0.02; MeO, 175-80.degree./ 0.05; MeS, 160.degree./0.01; F3C (prepd. from 4-F3CC6H4SH, b13 60-1.degree., which was prepd. from 4-F3CC6H4SO2Cl, b0.03 56-60.degree., m. 31-3.degree., which was obtained from 4-F3CC6H4NH2), 118-20.degree./ 0.02. XI (R = H, R' = Et) (78.0 g.) boiled 1 hr. with 13.0 g. NaOH in 78 ml. H2O and 53 ml. EtOH, the soln. concd. in vacuo, dild. with 200 ml. H2O, washed with 50 ml. CHCl3, acidified with 5N HCl, extd. with 1200 ml. CHCl3, the ext. washed with H2O, dried, concd. somewhat, and dild. with petr. ether gave XI (R = R' = H), m. 111-13.degree. (CHCl3-petr. ether). The following XI (R' = H) were prepd. similarly (R, m.p., and recrystn. solvent given): Cl, 134-5.degree., CHCl3-pentane; Me, 130-1.degree., EtOH-pentane: MeO, 124-6.degree., EtOH-pentane; MeS, 135-7.degree., EtOH-pentane; F3C, 125-8.degree., EtOH-pentane. XI (R = R' = H) (50.0 g.) heated 20 min. at 60.degree. with 200 g. SOCl2 and the product fractionated gave 2-(4-RC6H4SCH2)C6H4COCl (XII) (R = H), b0.1 165-7.degree.. Similarly was prepd. XII (R = Cl), b0.1 178-80.degree.. Method A. XII (R = H) (10.0 g.) in 70 ml. CS2 added dropwise during 30 min. to 10.0 g. AlCl3 suspended in 30 ml. boiling CS2, after 15 hrs. the CS2 removed, the residue treated with 50 g. ice and 15 ml. concd. HCl under cooling, extd. with 100 ml. Et2O, the ext. washed with 30 ml. 2N NaOH and with H2O until neutral, dried, concd., the crude product boiled in EtOH with C, and the EtOH soln. concd. deposited 11-oxo deriv. (XIII) of II, m. 84-6.degree. (EtOH); better yields were obtained by method B. Method B. To 207 ml. 85% H3PO4 was added 300 g. P2O5 at 80-100.degree. with stirring, the polyphosphoric acid mixt. kept at 100.degree., treated during 10 min. with 105.0 g. XI (R = Me, R' = H), stirred 75 min. at 100.degree., poured while hot onto 1 kg. ice with stirring, treated with 600 ml. C6H6, filtered through diatomaceous earth, the C6H6 layer in the filtrate sepd., the aq. layer extd. twice with 200 ml. C6H6, the combined C6H6 solns. extd. washed with 3 100-ml. portions 2N NaOH and with H2O until neutral, dried, concd., the residue dissolved in boiling EtOH, the soln. treated with C, and cooled to give 2-Me deriv. of XIII, m. 121-2.degree. (EtOH). Method C. XI (R = MeO, R' = H) (100.0 g.) added to 300 g. P2O5 and 200 ml. 85% H3PO4 in 2 l. PhMe at the b.p. with stirring, the mixt. heated 17 hrs., the PhMe soln. decanted while hot, the residue extd. with 4 1-l. portions boiling PhMe, the combined PhMe solns. washed with 11.2N NaOH and with H2O until neutral, dried, concd. in vacuo, the residue dissolved in boiling EtOH, the soln. treated with C, and cooled gave 2-MeO deriv. of XIII, m. 94-6.degree.. The following 2-substituted derivs. of XIII were also prepd. (2-substituent, method, and

m.p. given): Cl (XIV), B, 134-6.degree. (EtOH); MeS, C, 92-4.degree. (EtOH); F3C, B, 116-19.degree.. Iodine-activated Mg (1.1 g.) covered with a little tetrahydrofuran, treated with 0.1 ml. (BrCH2)2, when the reaction commenced the mixt. treated dropwise with 5.4 g. Me2N(CH2)3Cl in 10 ml. tetrahydrofuran in such a manner that the solvent boiled, refluxed 2 hrs., treated during 10 min. with 5.2 g. XIV in 15 ml. tetrahydrofuran, boiled and stirred 10 min., cooled, poured into 100 ml. H2O contg. 15 g. NH4Cl, treated with 100 ml. Et2O, filtered through diatomaceous earth, the Et2O layer in the filtrate sepd., the aq. layer extd. with 3 50-ml. portions Et2O, the combined Et2O solns. washed with H2O, dried, evapd., the oily residue dissolved in 10 ml. Me2CO, and the soln. kept gave 2-chloro-11-(3-dimethylaminopropyl)-11-hydroxy-6,11-dihydrodibenzo[b,e]thiepin {XV [R = Cl, R' = Me2N(CH2)3]} (XVa), m. 154-5.degree. (EtOH-pentane). XVa (5.0 g.) in 50 ml. AcOH boiled 1 hr. with 20 ml. concd. HCl, evapd. in vacuo (15 mm.), the residue made alk. with 2N NaOH, extd. with 3 50-ml. portions CHCl3, the combined exts. washed with H2O, dried, and evapd. gave 2-chloro-11-(3-dimethylamino-propylidene)-6,11-dihydrodibenzo[b,e]thiepin [XVI (R = Cl, R' = Me2NCH2-CH2CH)], oil; oxalate m. 215-16.degree. (EtOH). The following addnl. XV were prepd. (R, R', and m.p. given): H, 1-methyl-4-piperidyl, 184-7.degree.; H, 2-(1-methyl-2-piperidyl)-ethyl, 175-84.degree.; H, Me2N(CH2)3, 130-2.degree.; H, Et2N(CH2)3, 105-7.degree.; H, 3-(1-piperidyl)propyl, 190-2.degree.; H, 3-(1-morpholinyl)-propyl, 175-7.degree.; H, 3-(1-morpholinyl)-2-methylpropyl, 163-5.degree.; H. 1-methyl-3-piperidylmethyl, 170-5.degree.; H, 3-(1-piperidyl)-2-methylpropyl, 187-9.degree.; H, 1-methyl-3-pyrrolidylmethyl, -- (b0.15 200.degree.); H, 2-(1-methyl-2-pyrrolidyl)ethyl, 192-200.degree. and 116-20.degree. (2-isomers were isolated, in all other cases only 1 isomer was isolated); Cl, 1-methyl-4-piperidyl, 182-4.degree.; Cl, 3-(1-piperidyl)propyl, 195-7.degree.; Cl, 2-(1-methyl-2-piperidyl)ethyl, oil; Me, 1-methyl-4-piperidyl, 181-3.degree.; Me, Me2N(CH2)3, 139-42.degree.; MeS, 1-methyl-4-piperidyl, 178-80.degree.; MeS, Me2N(CH2)3, 137-8.degree.; MeO, 2-(1-methyl-2-piperidyl)ethyl, 141-2.degree.; MeO, Me2N(CH2)3, 123-5.degree.; MeO, 1-methyl-4-piperidyl, 182-5.degree.. The XV were not tested since previous experiences had shown them to have only slight activity. The following XVI were prepd. and tested [R, R', m.p., % histamine inhibition (thenalidine = 100%) (effective concn.: 5 .times. 10-8), % acetylcholine inhibition (atropine = 100%) (effective concn.: 1 .times. 10-9) given]: H, 1-methyl-4-piperidylidene (XVII), -- [HBr salt m. 265-70.degree. (decompn.)], 200, 33; H, 2-(1-methyl-2-piperidyl)ethylidene, -- [HBr salt m. 210-17.degree. (decompn.)], --, --; H, Me2NCH2CH2CH, -- (oxalate m. 167-9.degree.), 25, 10; H, Et2NCH2CH2CH, -- (oxalate m. 174-6.degree.), 33, 5; H, 3-(1-piperidyl)propylidene, -- (fumarate m. 193-7.degree.), 33,5; H, 3-(1-morpholinyl)propylidene, -- (fumarate m. 165-8.degree.), 50, 1.7; H, 3-(1-morpholinyl)-2-methyl-propylidene, -- (fumarate m. 182-5.degree.), 3.3, 0.5; H, 1-methyl-3-piperidylmethylene, -- (fumarate m. 240-2.degree.), 17, 10; H, 3-(1-piperidyl)-2-methylpropylidene, -- (oxalate m. 187-9.degree.), 10, 0.17; H, 1-methyl-3-pyrrolidylmethylene, -- (fumarate m. 213-15.degree.), 200, 17; 2-(1-methyl-2-pyrrolidyl)ethylidene, -- (oxalate m. 150-3.degree.), 400, 33; Cl, 1-methyl-4-piperidylidene, 161-4.degree., 200, 20; Cl, Me2NCH2CH2CH, -- (oxalate m. 215-16.degree.), 100, 2; Cl, 3-(1-piperidyl)propylidene, -- (fumarate m. 240-5.degree.), 7, 1.7; Cl, 2-(1-methyl-2-piperidyl)ethylidene, -- [HBr salt m. 245-60.degree. (decompn.)], 50, 6.5; Me, 1-methyl-4-piperidylidene, -- (HBr salt m. 294-7.degree.), 67, 3.3; Me, Me2NCH2CH2CH, -- (oxalate m. 189-92.degree.), 67, 3.3; MeS, 1-methyl-4-piperidylidene, 154-5.degree., 100, 4; MeS, Me2NCH2CH2CH, -- (oxalate m. 180-5.degree.), 100, 1.3; MeO, 2-(1-methyl-2-piperidyl)ethylidene, -- (HCl salt m. 204-11.degree.), 50, 5; MeO, Me2NCH2CH2CH, -- (oxalate m. 187-9.degree.), 100, 1.3; MeO, 1-methyl-4 piperidylidene, 120-1.degree., 100, 10. The I series showed weak activity as follows [compd., % histamine inhibition (thenalidene 100%), and % acetylcholine inhibition (atropine = 100%)given]: VII, 2, 6; VIII, 1, 7;

IX, 18, 2. The pharmacol. properties of XVII.HBr were more fully investigated. The antihistamine action of XVII.HBr was appreciably more pronounced in whole animal than in the in vitro studies. Thus 10-100 .gamma. XVII.HBr/kg. intravenously was able to arrest the blood pressure lowering effect of histamine in anesthetized cats. Subcutaneous doses of 0.15-0.3 mg. XVII. HBr/kg. prevented up to 50% the lethal and bronchoconstrictor action of histamine in guinea pigs. In these investigations in whole animals XVII.HBr was 20-30 times more effective than thenalidine. XVII.HBr also showed strong serotonin inhibiting action in the isolated rat uterus. It lacked any appreciable sedative effects.

L5 ANSWER 11 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1961:111931 CAPLUS
DN 55:111931
OREF 55:21040b-i,21041a-f
TI 2-Benzylthiobenzamides with antifungal activity
AU Gialdi, F.; Ponci, R.; Baruffini, A.
CS Univ. Pavia, Italy
SO Farmaco (Pavia), Ed. sci. (1960), 15, 856-82
DT Journal
LA Unavailable
AB 2-(Benzylthio)benzoic acid (24.4 g.) in 240 cc. C6H6 treated with 24 g. SOCl2, refluxed 2 hrs., treated with 240 cc. ligroine, cooled, and filtered yielded 85-90% 2-(benzylthio)benzoyl chloride (I), m. 121-2.degree.. I(1 g.) boiled 1 hr. with 7 g. anhyd. MeOH gave Me 2-(benzylthio)benzoate (II), m. 67.degree.. I (2.6 g.) in 40 cc. dioxane basified with NH3 gas, dild. with 120 cc. ice H2O, neutralized with AcOH, the ppt. filtered off, washed with H2O, and crystd. from EtOH yielded 87% 2-(benzylthio)benzamide (III), m. 154-5.degree.. III was obtained also from thiosalicylamide and benzyl chloride. Aniline (0.04 mole) in 30 cc. dioxane, treated dropwise with 0.02 mole I in 70 cc. dioxane, heated 30 min. at 50-60.degree., cooled, dild. with 150 cc. H2O, acidified with HCl, the soln. filtered and the ppt. crystd. from EtOH yielded 2-(benzylthio)benzanilide (IV), m. 122.degree.. Similarly N-butyl-2-(benzylthio)benzamide (V), m. 91-2.degree., was prepd. N-(Benzyl)thiosalicylamide (VI), m. 110.degree., was synthesized by treating 5 g. bis(benzylamide) of 2,2'-dicarboxydiphenyl disulfide (VII) in 50 cc. EtOH with 5 cc. concd. HCl and 6 g. Zn. VII was prepd. by oxidn. with 0.5% H2O2 of VI in NaOH. VI (0.5 g.), treated with a stoichiometric amt. of 0.5N NaOH and 0.25 g. PhCH2Cl in 10 cc. EtOH, the mixt. heated 15 min. at 50.degree. and cooled, yielded 0.3 g. 2-benzylthio-N-benzylbenzamide (VIII), m. 102-3.degree.. The hydrolysis of VIII with 10% NaOH gave 2-(benzylthio)benzoic acid, m. 189.degree.. VI and VII in EtOH refluxed 4 hrs. with Raney Ni gave N-benzylbenzamide. VIII was obtained also from VII by condensing with PhCH2Cl with K2CO3 and refluxing 15 hrs. with PhCH2NH2. By the same method as for IV, the N,N-diethyl-2-(benzylthio)benzamide (IX), m. 81.degree. was prepd. N-[2-(Benzylthio)benzoyl]morpholine (X), m. 114.degree., and N-[2-(benzylthio)benzoyl] piperidine, m. 117-18.degree., were synthesized by the same method as for V. II, refluxed 2 hrs. with 7 cc. 95% hydrazine gave 2-(benzylthio)benzohydrazide, m. 164.degree.. Me thiosalicylate (16.8 g.) in 150 cc. EtOH, treated with 16.1 g. p-chlorobenzyl chloride (XI) with 6.9 g. K2CO3, the mixt. refluxed 1 hr., cooled, the soln. poured into 2 vols. ice H2O, and the ppt. filtered off and crystd. from EtOH yielded Me p-chlorobenzylthiobenzoate (XII), m. 102-3.degree.. 2-(4-Chlorobenzylthio)benzoic acid (XIII), m. 216-17.degree., was obtained by condensing thiosalicylic acid (XIV) and XI, in the presence of K2CO3 or boiling XII with concd. HCl. XIV (3.08 g.) in 30 cc. EtOH treated with 6.44 g. XI with 2.7 g. K2CO3, the mixt. refluxed 1 hr., the suspension dild. twice with ice H2O, filtered and the ppt. crystd. from acetone yielded 4-chlorobenzyl 2-(4 chlorobenzylthio)benzoate (XV), m. 166-7.degree.. XV boiled 5 hrs. with 1:1 EtOH- 10% NaOH gave XIII. 2-(4-Chlorobenzylthio)benzoyl chloride (XVI), m. 108-10.degree., was prepd. by the method as for I and the Me ester (XVII), m. 84.degree., was

obtained from XVI as for II. 2-(4-Chlorobenzylthiobenzamide (XVIII), m. 147-8.degree., 2-(4-chlorobenzylthio)benzanilide (XIX), m. 127-8.degree., and 2-(4-chlorobenzylthio)-N-butylbenzamide, m. 98-100.degree., were prepd. As for IV, 2-(4-chlorobenzylthio)-N-benzylbenzamide, m. 130.degree., and 2-(4-chlorobenzylthio)-N,N-diethylbenzamide, m. 76-7.degree., were obtained. N-[2-(4-Chlorobenzylthio)benzoyl] morpholine (XX), m. 68-9.degree., and N-[2-(4-chlorobenzylthio)benzoyl]piperidine (XXI), m. 72-4.degree., were synthesized. The hydrazide (XXII) of 2-(4-chlorobenzylthio)benzoic acid, m. 166.degree., was obtained by boiling 5 hrs. under pressure 5 g. XVII and 1.5 cc. 95% hydrazine. 2-(4-Methoxybenzylthio)benzoic acid (XXIII), m. 218-19.degree., was obtained. p-Methoxybenzyl alcohol (40 g.), cooled on ice, treated dropwise with stirring with 50 g. $SOCl_2$ during 20 min., the mixt. heated 1 hr. at 40.degree., cooled, treated with 2 g. $CaCO_3$ and 60 cc. anhyd. Et_2O, stirred several hrs., and finally kept 12 hrs. at room temp. yielded, after filtration and evapn. of Et_2O and $SOCl_2$, an oil, b5.0 98-102.degree., identified as p-methoxybenzyl chloride (XXIV). XXIII (30 g.) refluxed 1.5 hrs. with 45 cc. $SOCl_2$ yielded 2-(4-methoxybenzylthio)benzoyl chloride (XXV), m. 106-8.degree. (C_6H_6-petr. ether). This chloride with EtOH, as for II, gave Et 2-(4-methoxybenzylthio)benzoate, m. 100.degree.. Condensing XXIII with XXIV gave p-methoxybenzyl 2-(4-methoxybenzylthio)benzoate (XXVI), m. 114-15.degree.. XXVI, on hydrolysis, gave XXIII. NH_3 in 8 cc. dioxane, treated dropwise with 3 g. XXV in 10 cc. dioxane, the mixt. kept 3 hrs. at room temp., dild. with 40 cc. H_2O, neutralized with dil. HCl, and the ppt. filtered off and crystd. from EtOH yielded 2-(4-methoxybenzylthio)benzamide, m. 147.degree.. From XXV and aniline 2-(4-methoxybenzylthio)benzanilide, m. 135.degree., was obtained. Also prepd. were: 2-(4-methoxybenzylthio)-N-butylbenzamide, m. 87-90.degree.; 2-(4-methoxybenzylthio)-N-benzylbenzamide, m. 107-9.degree.; 2-(4-methoxybenzylthio)benzohydrazide (XXVIa), m. 143.degree.; 2-(4-nitrobenzylthio)benzoyl chloride, m. 128-9.degree.; Et 2-(4-nitrobenzylthio)benzoate (XXVII), m. 91.degree.; 2-(4-nitrobenzylthio)benzamide (XXVIII), m. 143-4.degree.; 2-(4-nitrobenzylthio)benzanilide (XXIX), m. 116.degree.; 2-(4-nitrobenzylthio)-N-butylbenzamide (XXX), m. 87-9.degree.; 2-(4-nitrobenzylthio)-N-benzylbenzamide (XXXI), m. 140.degree.. XXVII (1.5 g.), refluxed 1 hr. with 3 cc. 95% hydrazine and the soln. neutralized with AcOH yielded 2-(4-nitrophenyl)-3-hydroxybenzothiophene (XXXII), m. 195.degree.. XXXII was also obtained by condensing XXVII with NaOMe. XXVII (5 g.) in 50 cc. 95% EtOH autoclaved with H at 50 atm. and 65.degree. with 0.3 g. Raney Ni 8 hrs. yielded Et 2-(4-aminobenzylthio)benzoate (XXXIII), m. 106.degree.. The acetyl deriv. (XXXIV), m. 158.degree., was obtained by refluxing XXXIII with AcOH in presence of a drop of AcCl. By this procedure, from XXVIII, 2-(4-aminobenzylthio)benzamide, m. 173.degree., was obtained; the Ac deriv., m. 262.degree., was synthesized by the same method as for XXXIV. The catalytic redn. of XXIX at 70 atm. yielded 2-(4-aminobenzylthio)benzanilide, m. 120-1.degree.; Ac deriv. m. 215.degree.. XXX and XXXI heated at 50.degree./50 atm. 5 hrs. gave 2-(4-aminobenzylthio)-N-butylbenzamide (XXXV), m. 92.degree. (Ac deriv. m. 209.degree.), and 2-(4-aminobenzylthio)-N-benzylbenzamide (XXXVI), m. 119-20.degree. (Ac deriv. m. 213.degree.). XXXIII (1 g.) in 10% dioxane with 5 g. 95% hydrazine, and the mixt. refluxed 3 hrs. gave 2-(4-aminobenzylthio)benzohydrazide, m. 197-8.degree. (EtOH). The 2-benzylthiobenzamides prepd. were tested in vitro on Candida albicans ATCC 10231 and Trichophyton mentagrophytes ATCC 8757. All the substances proved to be inactive within the limits of soly. (between 5 and 50 .gamma./cc.) or at the max. concn. of 100 .gamma./cc. against the yeast-like microorganism. Against T. mentagrophytes IX, XX, XXI, XXII, XXVIa, XXXV, and XXXVI proved to be active. The same substances were tested in vitro against Madurella grisea, Microsporum audouini, Stemphylium sarciniforme, Aspergillus fumigatus, Cryptococcus neoformans, and Nocardia asteroides and good antifungal activity was found.

L5 ANSWER 12 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1958:11324 CAPLUS
DN 52:11324
OREF 52:2069i,2070a-c
TI Sulfur-containing compounds
IN Stevenson, Herbert A.; Greenwood, Douglas; Higgons, Dennis J.; Cranham, John E.
PA Boots Pure Drug Co. Ltd.
DT Patent
LA Unavailable
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 780520		19570807	GB	

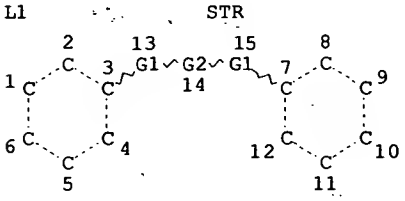
PI GB 780520 19570807 GB

AB New benzyl phenyl sulfides have been synthesized which are valuable for the control of Tetranychide (Red Spider mites), e.g., Tetranychus telarius L. and Metatetranychus ulmi Koch. A mixt. of 8.5 g. p-ClC6H4SH, 10 g. of p-NCC6H4CH2Br, 1.4 g. Na, and 100 cc. alc. was refluxed two hrs., cooled, and dild. with 500 cc. H2O, and the ppt. filtered off to give p-chlorophenyl p-cyanobenzyl sulfide, m. 76-7.degree. (alc.). The following compds. were prepd. in a similar way: p-cyanobenzyl phenyl sulfide (m. 73-4.degree.), p-cyanobenzyl p-fluorophenyl sulfide (m. 48-9.degree.), .omicron.-(p-cyanobenzylthio)benzoic acid (m. 220.degree.), and .omicron.-(p-chlorobenzylthio)benzoic acid (m. 222.degree.). By stirring 16.8 g. .omicron.-(p-chlorobenzylthio)benzyl chloride with 300 cc. aq. NH3, .omicron.-(p-chlorobenzylthio)benzamide, m. 144-5.degree., was prepd. .omicron.-(p-cyanobenzylthio)benzamide (m. 155-6.degree.) and .omicron.-(benzylthio)benzamide, m. 152-3.degree., were similarly prepd. A prepn. of p-chlorobenzyl .omicron.-cyanophenyl sulfide was made from 2.21 g. POCl3 in 10 cc. dry C5H5N and 2.0 g. .omicron.-(p-chlorobenzylthio)benzamide, m. 55-6.degree.. Benzyl .omicron.-cyanophenyl sulfide (m. 65-6.degree.) and p-cyanobenzyl .omicron.-cyanophenyl sulfide, m. 109-10.degree. were prepd. in the same manner. Et .omicron.-(p-chlorobenzylthio)benzoate (m. 87.degree.) was prepd. from the acid and EtOH in the presence of H2SO4. The Me ester, m. 102.degree., was prepd.

L5 ANSWER 13 OF 13 CAPLUS COPYRIGHT 2003 ACS
AN 1955:53500 CAPLUS
DN 49:53500
OREF 49:10267g-i,10268a-d
TI Derivatives of 5-o-mercaptophenyl-3-methyl-1-phenylpyrazole
AU Barry, W. J.; Finar, I. L.
CS Northern Polytech., London
SO J. Chem. Soc. (1954) 138-40
DT Journal
LA Unavailable
AB Some new (.omicron.o-substituted-phenyl)pyrazoles are prepd. in which ring-closure is effected between substituent groups to form a new polycyclic system. .omicron.-PhCH2SC6H4CO2H heated 0.5 hr. with 2-3 moles SOCl2 gives 60% of the acid chloride (I), m. 121-2.degree.. I (1.1 moles) and 1 mole AcCH2CO2Et in NaOEt yields 27% PhCH2SC6H4CO2Et (II), m. 68.degree., alone or mixed with II prepd. by heating an excess of I with EtOH. Acidification of the filtrate gives 73% of the diketo ester (III); Cu deriv., bluish-green crystals from CHCl3-ligroine. III (1 mole) heated 2 hrs. at 100.degree. with 1.1 moles PhNHNH2 in HOAc affords 83% Et ester (IV), m. 121-2.degree., of 5-.omicron.-mercaptophenyl-3-methyl-1-phenyl-4-pyrazolecarboxylic acid (V), m. 236.degree. (decompn.). V heated at 250-5.degree. for 1-1.5 hrs. decarboxylates to yield 60% 5-.omicron.-benzylthiophenyl-3-methyl-1-phenylpyrazole (VI), m. 110.degree.. Cl passed 0.5 hr. through 40 g. IV, in 1 l. HOAc and 25 ml. H2O at 0.degree. and the soln. set aside 10 min. gives 36 g.Et5-.omicron.-chlorosulfonylphenyl-3-methyl-1-phenyl-4-pyrazolecarboxylate (VII), m. 155-6.degree.; anilide, m. 157.5.degree.. Similar chlorination of either V or VI gives 80% yield

4-chloro-5-.omicron.-chlorosulfonylphenyl-3-methyl-1-phenylpyrazole (VIII), m. 145.degree.. VII (12 g.) kept 12 hrs. at room temp. with 10 g. Zn dust, 100 ml. HOAc, and 20 ml. concd. HCl, 20 ml. more HCl added, the soln. left 1 hr. longer, then treated with H2O to turbidity, gave next morning 9.5 g. Et 3-methyl-1-phenyl-5-.omicron.-sulfinophenyl-4-pyrazolecarboxylate (IX), m. 186.degree. (sealed tube), hydrolyzed with 10% KOH-EtOH in 0.5 hr. to 82% of the corresponding carboxylic acid (X), m. 244.degree. (sealed tube). IX (10 g.) refluxed in 100 ml. HOAc and 100 ml. 3N H2SO4 and treated portionwise with 25 g. Zn dust during 1.5 hrs. gives 2-3 g. 5-.omicron.-mercaptophenyl-3-methyl-1-phenyl-4-pyrazolecarboxylic acid lactone (XI), m. 208-10.degree., also prepd. by the addn. of concd. HCl to a refluxing soln. of IX in HOAc with granulated Zn. XI refluxed several min. with 20% KOH-EtOH and acidified gives the thiol (XII), m. 158-60.degree., frothing and resolidifying to m. again at 208-10.degree., which forms white and yellow ppts. with HgCl2 and Pb(OAc)2, resp. The addn. of concd. HCl to XII in refluxing EtOH gives XI. XII warmed with 10% Na2CO3 soln. and PhCH2Cl forms 5-.omicron.-benzylthiophenyl-3-methyl-1-phenyl-4-pyrazolecarboxylic acid (XIII), m. 235-6.degree.. The Et ester of XIII (7.5 g.) heated 15 min. with 100 ml. 10% KOH-EtOH gives 5.2 g. free acid, which, heated 1.5 hrs. at 250-70.degree., yields 5-.omicron.-benzylsulfonylphenyl-3-methyl-1-phenylpyrazole (XIV), m. 182-3.degree.. VI (0.75 g.) in 10 ml. HOAc heated 1 hr. at 100.degree. with 3 ml. 30% H2O2 yields 0.5 g. XIV. XIV (1 g.) heated 35 hrs. with 25 g. 5% Na-Hg in 25 ml. EtOH gives .omicron.(3-methyl-1-phenyl-5-pyrazolyl)benzenesulfinic acid (XV), characterized by conversion with BzCl in excess K2CO1, to the sulfone (XVI), m. 180-2.degree.. The Et ester of XIII (1 g.) refluxed 9 hrs. with 10 g. Raney Ni in 50 ml. EtOH gives Et 1,5-diphenyl-4-pyrazolecarboxylate (XVII), m. 119-21.degree.. The identity of XVII is confirmed by hydrolysis to the acid, m. 205.degree..

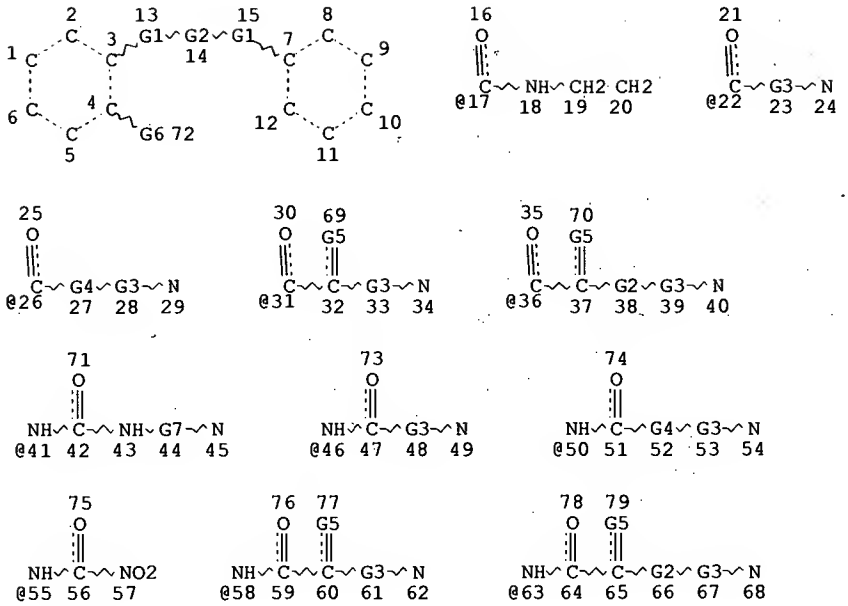
```
=> fil reg; d stat que l18; fil hcapl; d que nos 120; fil uspatf; d que nos l22; dup rem
120,122
FILE 'REGISTRY' ENTERED AT 12:28:47 ON 09 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2003 American Chemical Society (ACS)

Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES:    8 APR 2003  HIGHEST RN 502421-05-8
DICTIONARY FILE UPDATES:   8 APR 2003  HIGHEST RN 502421-05-8

TSCA INFORMATION NOW CURRENT THROUGH MAY 20, 2002

  Please note that search-term pricing does apply when
  conducting SmartSELECT searches.

Crossover limits have been increased.  See HELP CROSSOVER for details.

Experimental and calculated property data are now available.  See HELP
PROPERTIES for more information.  See STNote 27, Searching Properties
in the CAS Registry File, for complete details:
http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

L1                STR
```



full file search done on this structure

```
REP G1=(0-5) CH2
VAR G2=O/S/N
NODE ATTRIBUTES:
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS  15

STEREO ATTRIBUTES: NONE
L3        32974 SEA FILE=HCAPLUS ABB=ON  (CA OR CALCIUM)(L)CHANNEL/OBI
L4              SEL  L3 1- RN :   50639 TERMS  (TERM LIMIT EXCEEDED)
L5        50630 SEA FILE=REGISTRY ABB=ON  L4
L6              SEL  L3 4002- RN :   50302 TERMS  (TERM LIMIT EXCEEDED)
L7        49953 SEA FILE=REGISTRY ABB=ON  L6
L8              SEL  L3 25337- RN :   18709 TERMS
L9        18569 SEA FILE=REGISTRY ABB=ON  L8
L10      102016 SEA FILE=REGISTRY ABB=ON  (L5 OR L7 OR L9)
L12        3693 SEA FILE=REGISTRY SUB=L10 SSS FUL L1
L15             STR
```

Searched by Barb O'Bryen, STIC 308-4291



REP G1=(0-5) CH2
VAR G2=O/S/N
REP G3=(1-6) CH2
VAR G4=O/S/N
VAR G5=O/S
VAR G6=17/22/26/31/36/41/46/50/55/58/63
REP G7=(2-5) CH2
NODE ATTRIBUTES:
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

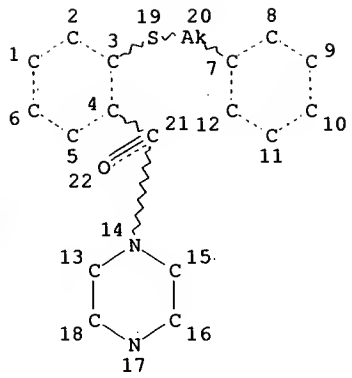
GRAPH ATTRIBUTES:
RSPEC I
NUMBER OF NODES IS 79

STEREO ATTRIBUTES: NONE
L16 STR

claim 21 ~~20~~

subset search done looking for this structure or structure on following page

Searched by Barb O'Bryen, STIC 308-4291



claim 25

NODE ATTRIBUTES:
CONNECT IS E2 RC AT 20
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 22

STEREO ATTRIBUTES: NONE

L18 [redacted]

100.0% PROCESSED 2478 ITERATIONS [redacted]
SEARCH TIME: 00.00.01

[redacted] ENTERED AT 12:28:48 ON 09 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

FILE COVERS 1907 - 9 Apr 2003 VOL 138 ISS 15
FILE LAST UPDATED: 8 Apr 2003 (20030408/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

L1 STR

Searched by Barb O'Bryen, STIC 308-4291

```
L3        32974 SEA FILE=HCAPLUS ABB=ON  (CA OR CALCIUM)(L)CHANNEL/OBI
L4              SEL  L3 1- RN :   50639 TERMS  (TERM LIMIT EXCEEDED)
L5        50630 SEA FILE=REGISTRY ABB=ON  L4
L6              SEL  L3 4002- RN :   50302 TERMS  (TERM LIMIT EXCEEDED)
L7        49953 SEA FILE=REGISTRY ABB=ON  L6
L8              SEL  L3 25337- RN :   18709 TERMS
L9        18569 SEA FILE=REGISTRY ABB=ON  L8
L10      102016 SEA FILE=REGISTRY ABB=ON  (L5 OR L7 OR L9)
L12        3693 SEA FILE=REGISTRY SUB=L10 SSS FUL L1
L15             STR
L16             STR
L18            11 SEA FILE=REGISTRY SUB=L12 SSS FUL (L15 OR L16)
L20             2 SEA FILE=HCAPLUS ABB=ON L18
```

FILE 'USPATFULL' ENTERED AT 12:28:48 ON 09 APR 2003
CA INDEXING COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)

FILE COVERS 1971 TO PATENT PUBLICATION DATE: 8 Apr 2003 (20030408/PD)
FILE LAST UPDATED: 8 Apr 2003 (20030408/ED)
HIGHEST GRANTED PATENT NUMBER: US6546558
HIGHEST APPLICATION PUBLICATION NUMBER: US2003066115
CA INDEXING IS CURRENT THROUGH 8 Apr 2003 (20030408/UPCA)
ISSUE CLASS FIELDS (/INCL) CURRENT THROUGH: 8 Apr 2003 (20030408/PD)
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Feb 2003
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Feb 2003

>>> USPAT2 is now available. USPATFULL contains full text of the <<<
>>> original, i.e., the earliest published granted patents or <<<
>>> applications. USPAT2 contains full text of the latest US <<<
>>> publications, starting in 2001, for the inventions covered in <<<
>>> USPATFULL. A USPATFULL record contains not only the original <<<
>>> published document but also a list of any subsequent <<<
>>> publications. The publication number, patent kind code, and <<<
>>> publication date for all the US publications for an invention <<<
>>> are displayed in the PI (Patent Information) field of USPATFULL <<<
>>> records and may be searched in standard search fields, e.g., /PN, <<<
>>> /PK, etc. <<<

>>> USPATFULL and USPAT2 can be accessed and searched together <<<
>>> through the new cluster USPATALL. Type FILE USPATALL to <<<
>>> enter this cluster. <<<
>>> <<<
>>> Use USPATALL when searching terms such as patent assignees, <<<
>>> classifications, or claims, that may potentially change from <<<
>>> the earliest to the latest publication. <<<

This file contains CAS Registry Numbers for easy and accurate
substance identification.

```
L1              STR
L3        32974 SEA FILE=HCAPLUS ABB=ON  (CA OR CALCIUM)(L)CHANNEL/OBI
L4              SEL  L3 1- RN :   50639 TERMS  (TERM LIMIT EXCEEDED)
L5        50630 SEA FILE=REGISTRY ABB=ON  L4
L6              SEL  L3 4002- RN :   50302 TERMS  (TERM LIMIT EXCEEDED)
L7        49953 SEA FILE=REGISTRY ABB=ON  L6
L8              SEL  L3 25337- RN :   18709 TERMS
L9        18569 SEA FILE=REGISTRY ABB=ON  L8
L10      102016 SEA FILE=REGISTRY ABB=ON  (L5 OR L7 OR L9)
L12        3693 SEA FILE=REGISTRY SUB=L10 SSS FUL L1
L15             STR
```

Searched by Barb O'Bryen, STIC 308-4291

L16 STR
L18 11 SEA FILE=REGISTRY SUB=L12 SSS FUL (L15 OR L16)
[redacted]

FILE 'HCAPLUS' ENTERED AT 12:28:48 ON 09 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)

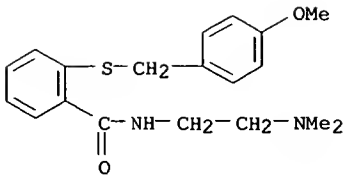
FILE 'USPATFULL' ENTERED AT 12:28:48 ON 09 APR 2003
CA INDEXING COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)
PROCESSING COMPLETED FOR L20
PROCESSING COMPLETED FOR L22
[redacted]
ANSWERS '1-2' FROM FILE HCAPLUS
ANSWER '3' FROM FILE USPATFULL

[redacted]

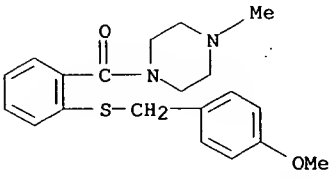
L24 ANSWER 1 OF 3 HCAPLUS COPYRIGHT 2003 ACS DUPLICATE 1
ACCESSION NUMBER: 2002:638285 HCAPLUS
DOCUMENT NUMBER: 137:185512
TITLE: Preparation of S-benzylthiosalicylamides and analogs as calcium channel blockers
INVENTOR(S): Mehanna, Ahmed S.; Kim, Jinyung T.
PATENT ASSIGNEE(S): Massachusetts College of Pharmacy, USA
SOURCE: U.S. Pat. Appl. Publ., 31 pp., Division of U. S. Ser. No. 982,953.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2002115655	A1	20020822	US 2001-998623	20011031
US 6541479	B1	20030401	US 1997-982953	19971202

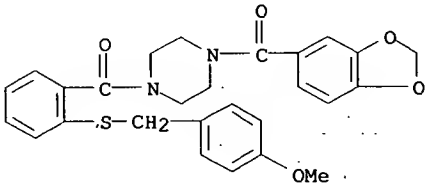
PRIORITY APPLN. INFO.: US 1997-982953 A3 19971202
OTHER SOURCE(S): MARPAT 137:185512
AB R1ZR2 [I; R1,R2 = (un)substituted (hetero)aryl; Z = (CH2)mZ1(CH2)n; Z1 = O, S, N (sic); m,n = 0-5] were prepd. Thus, 2-(HS)C6H4CO2H was thioetherified by 4-(MeO)C6H4CH2Cl and the product amidated by 1-methylpiperazine to give 4-(MeO)C6H4CH2SC6H4(COR)-2 (R = 4-methylpiperazino). Data for biol. activity of I were given.
IT **449174-74-7P 449174-76-9P 449174-78-1P**
449174-80-5P 449174-82-7P 449174-84-9P
449174-86-1P 449174-88-3P
RL: PAC (Pharmacological activity); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)
(prepn. of S-benzylthiosalicylamides and analogs as calcium channel blockers)
RN 449174-74-7 HCAPLUS
CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[[(4-methoxyphenyl)methyl]thio]- (9CI) (CA INDEX NAME)



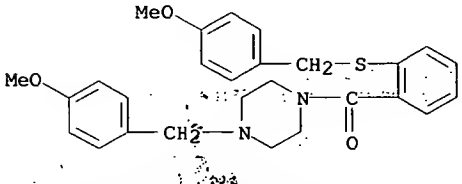
RN 449174-76-9 HCAPLUS
CN Piperazine, 1-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]-4-methyl- (9CI) (CA INDEX NAME)



RN 449174-78-1 HCAPLUS
CN Piperazine, 1-(1,3-benzodioxol-5-ylcarbonyl)-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)

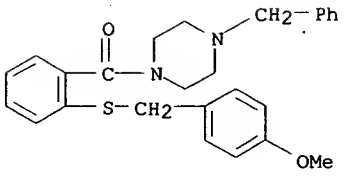


RN 449174-80-5 HCAPLUS
CN Piperazine, 1-[(4-methoxyphenyl)methyl]-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)

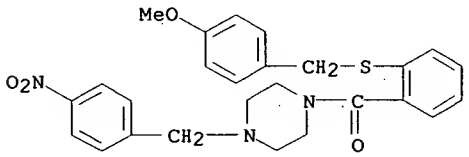


RN 449174-82-7 HCAPLUS
CN Piperazine, 1-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]-4-(phenylmethyl)- (9CI) (CA INDEX NAME)

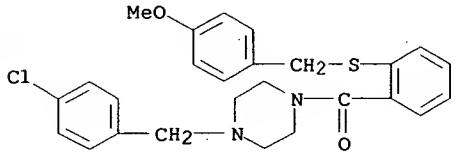
Searched by Barb O'Bryen, STIC 308-4291



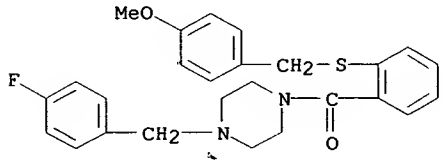
RN 449174-84-9 HCAPLUS
CN Piperazine, 1-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]-4-[(4-nitrophenyl)methyl]- (9CI) (CA INDEX NAME)



RN 449174-86-1 HCAPLUS
CN Piperazine, 1-[(4-chlorophenyl)methyl]-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)



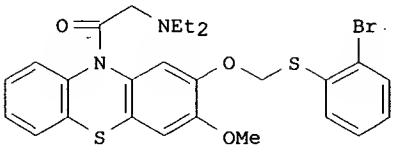
RN 449174-88-3 HCAPLUS
CN Piperazine, 1-[(4-fluorophenyl)methyl]-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)



L24 ANSWER 2 OF 3 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1995:15500 HCAPLUS
DOCUMENT NUMBER: 122:56006
TITLE: Regioselective cleavage reaction of the aromatic methylenedioxy ring. VI. Synthesis of phenothiazine analogs by using the cleavage reaction with sodium methoxide-thiols in dimethyl sulfoxide and evaluation of their biological activities
AUTHOR(S): Imakura, Yasuhiro; Konishi, Tatsuya; Uchida, Kazuiti;

Searched by Barb O'Bryen, STIC 308-4291

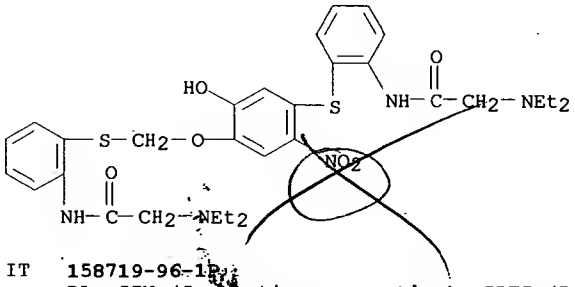
Sakurai, Hiromu; Kobayashi, Shigeru; Haruno, Akihiro; Tajima, Kiyotaka; Yamashita, Shinsuke
CORPORATE SOURCE: Fac. Sci., Naruto Univ. Educ., Naruto, 772, Japan
SOURCE: Chemical & Pharmaceutical Bulletin (1994), 42(3), 500-11
CODEN: CPBTAL; ISSN: 0009-2363
DOCUMENT TYPE: Journal
LANGUAGE: English
GI



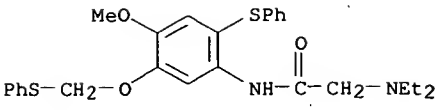
I

AB The reactions of arom. methylenedioxy compds. contg. electron-withdrawing groups with Na methoxide-thiols in DMSO gave 3- and 4-hydroxybenzene derivs. in good yield by regioselective attack of the thiolate ions on the methylenedioxy ring. The formation mechanism and the reactivity of thiolate ions in the cleavage reaction of the methylenedioxy ring are discussed. Various biol. active compds., were prepd. from the 4-hydroxybenzene derivs. and their Ca2+ antagonistic activities were evaluated. Among these compds., 2-(2-bromophenylthiomethoxy)-10-(2-diethylaminoacetyl)-3-methoxyphenothiazine (I) showed the most potent Ca2+ antagonistic activity. Biol. activity could be conveniently evaluated by measurement of the peak height of the vanadyl ion (+4 oxidn. ion) signal produced by redox reaction between the phenothiazine derivs. and vanadate ion (+5 oxidn. ion) with ESR spectroscopy.

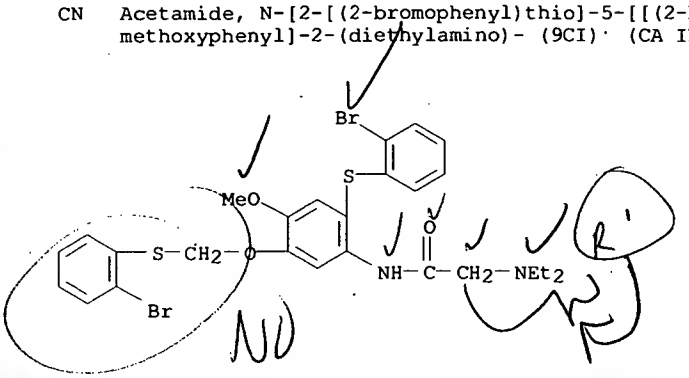
IT **158719-93-8**
RL: RCT (Reactant); RACT (Reactant or reagent)
(calcium antagonist)
RN 158719-93-8 HCAPLUS
CN Acetamide, 2-(diethylamino)-N-[2-[[[4-[[2-[[(diethylamino)acetyl]amino]phenyl]thio]-2-hydroxy-5-nitrophenoxy]methyl]thio]phenyl]- (9CI) (CA INDEX NAME)



IT **158719-96-1P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
RN 158719-96-1 HCAPLUS
CN Acetamide, 2-(diethylamino)-N-[4-methoxy-2-(phenylthio)-5-[(phenylthio)methoxy]phenyl]- (9CI) (CA INDEX NAME)



IT **158719-87-0P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of, as calcium antagonist)
RN 158719-87-0 HCAPLUS
CN Acetamide, N-[2-[(2-bromophenyl)thio]-5-[[(2-bromophenyl)thio]methoxy]-4-methoxyphenyl]-2-(diethylamino)- (9CI) (CA INDEX NAME)



L24 ANSWER 3 OF 3 USPATFULL
ACCESSION NUMBER: 2003:89389 USPATFULL
TITLE: Calcium channel blockers
INVENTOR(S): Mehanna, Ahmed S., Sudbury, MA, United States
Kim, Jinyung T., Boston, MA, United States
PATENT ASSIGNEE(S): Massachusetts College of Pharmacy, Boston, MA, United States (U.S. corporation)

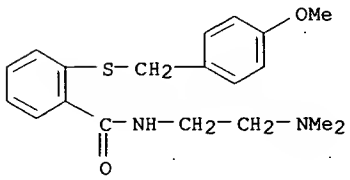
	NUMBER	KIND	DATE	
PATENT INFORMATION:	US 6541479	B1	20030401	
APPLICATION INFO.:	US 1997-982953		19971202	(8)

DOCUMENT TYPE: Utility
FILE SEGMENT: GRANTED
PRIMARY EXAMINER: Jones, Dwayne C.
LEGAL REPRESENTATIVE: Wolf, Greenfield & Sacks, P.C.
NUMBER OF CLAIMS: 16
EXEMPLARY CLAIM: 1
NUMBER OF DRAWINGS: 7 Drawing Figure(s); 4 Drawing Page(s)
LINE COUNT: 1668
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB The invention involves the identification of a family of compounds which block calcium channels. The compounds can be formulated in pharmaceutical carriers and administered to subjects. The compounds are useful for treating disorders associated with calcium channel activity, such as, cardiovascular diseases, for example hypertension, congestive heart failure, arrhythmia and angina.

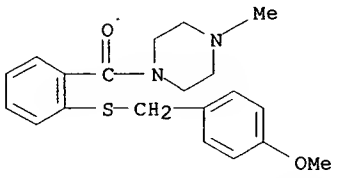
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **449174-74-7P 449174-76-9P 449174-78-1P**
449174-80-5P 449174-82-7P 449174-84-9P
449174-86-1P 449174-88-3P
(prepn. of S-benzylthiosalicylamides and analogs as calcium channel blockers)
RN 449174-74-7 USPATFULL

Searched by Barb O'Bryen, STIC 308-4291

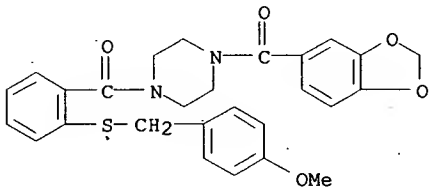
CN Benzamide, N-[2-(dimethylamino)ethyl]-2-[[(4-methoxyphenyl)methyl]thio]- (9CI) (CA INDEX NAME)



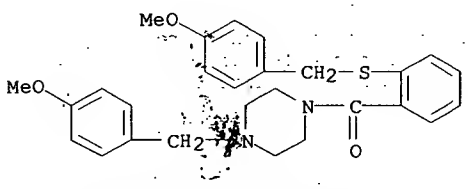
RN 449174-76-9 USPATFULL
CN Piperazine, 1-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]-4-methyl- (9CI) (CA INDEX NAME)



RN 449174-78-1 USPATFULL
CN Piperazine, 1-(1,3-benzodioxol-5-ylcarbonyl)-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)

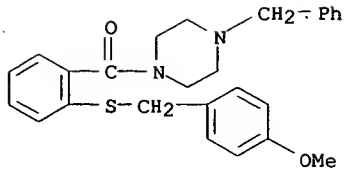


RN 449174-80-5 USPATFULL
CN Piperazine, 1-[(4-methoxyphenyl)methyl]-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)

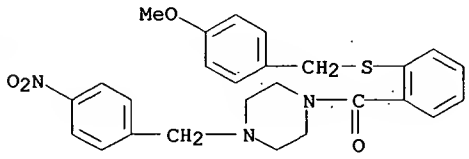


RN 449174-82-7 USPATFULL
CN Piperazine, 1-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]-4-(phenylmethyl)- (9CI) (CA INDEX NAME)

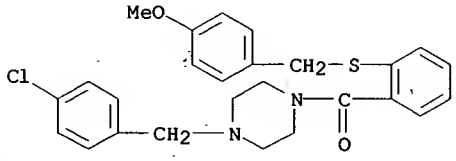
Searched by Barb O'Bryen, STIC 308-4291



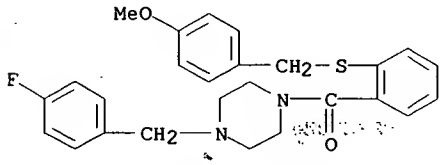
RN 449174-84-9 USPATFULL
CN Piperazine, 1-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]-4-[(4-nitrophenyl)methyl]- (9CI) (CA INDEX NAME)



RN 449174-86-1 USPATFULL
CN Piperazine, 1-[(4-chlorophenyl)methyl]-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)



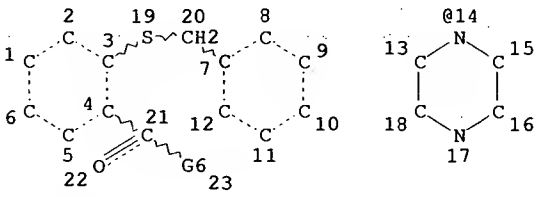
RN 449174-88-3 USPATFULL
CN Piperazine, 1-[(4-fluorophenyl)methyl]-4-[2-[[(4-methoxyphenyl)methyl]thio]benzoyl]- (9CI) (CA INDEX NAME)



Searched by Barb O'Bryen, STIC 308-4291

```
=> fil reg; d stat que l28
FILE 'REGISTRY' ENTERED AT 12:33:26 ON 09 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2003 American Chemical Society (ACS)

Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES:    8 APR 2003  HIGHEST RN 502421-05-8
DICTIONARY FILE UPDATES:   8 APR 2003  HIGHEST RN 502421-05-8

TSCA INFORMATION NOW CURRENT THROUGH MAY 20, 2002

  Please note that search-term pricing does apply when
  conducting SmartSELECT searches.

Crossover limits have been increased.  See HELP CROSSOVER for details.

Experimental and calculated property data are now available.  See HELP
PROPERTIES for more information.  See STNote 27, Searching Properties
in the CAS Registry File, for complete details:
http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf
```

L25 STR



claim 20 & 25

```
VAR G6=14/X
NODE ATTRIBUTES:
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS  23

STEREO ATTRIBUTES: NONE
L26  20 SEA FILE=REGISTRY SSS FUL L25

100.0% PROCESSED     64 ITERATIONS                    20 ANSWERS
SEARCH TIME: 00.00.01


=> fil hcaplus; d que nos l29; s l29 not l20
FILE 'HCAPLUS' ENTERED AT 12:35:38 ON 09 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)
```

Searched by Barb O'Bryen, STIC 308-4291

FILE COVERS 1907 - 9 Apr 2003 VOL 138 ISS 15
FILE LAST UPDATED: 8 Apr 2003 (20030408/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

L25 STR
L28 29 SEA FILE=REGISTRY SSS FUL L25
L29 26 SEA FILE=HCAPLUS ABB=ON L28
L30 26 L29 NOT L20 previously printed

=> fil uspatf; d que nos 130; s 130 not 122

USPATFULL ENTERED AT 12:35:39 ON 09 APR 2003
CA INDEXING COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)

FILE COVERS 1971 TO PATENT PUBLICATION DATE: 8 Apr 2003 (20030408/PD)
FILE LAST UPDATED: 8 Apr 2003 (20030408/ED)
HIGHEST GRANTED PATENT NUMBER: US6546558
HIGHEST APPLICATION PUBLICATION NUMBER: US2003066115
CA INDEXING IS CURRENT THROUGH 8 Apr 2003 (20030408/UPCA)
ISSUE CLASS FIELDS (/INCL) CURRENT THROUGH: 8 Apr 2003 (20030408/PD)
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Feb 2003
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Feb 2003

>>> USPAT2 is now available. USPATFULL contains full text of the <<<
>>> original, i.e., the earliest published granted patents or <<<
>>> applications. USPAT2 contains full text of the latest US <<<
>>> publications, starting in 2001, for the inventions covered in <<<
>>> USPATFULL. A USPATFULL record contains not only the original <<<
>>> published document but also a list of any subsequent <<<
>>> publications. The publication number, patent kind code, and <<<
>>> publication date for all the US publications for an invention <<<
>>> are displayed in the PI (Patent Information) field of USPATFULL <<<
>>> records and may be searched in standard search fields, e.g., /PN, <<<
>>> /PK, etc. <<<

>>> USPATFULL and USPAT2 can be accessed and searched together <<<
>>> through the new cluster USPATALL. Type FILE USPATALL to <<<
>>> enter this cluster. <<<
>>> <<<
>>> Use USPATALL when searching terms such as patent assignees, <<<
>>> classifications, or claims, that may potentially change from <<<
>>> the earliest to the latest publication. <<<

This file contains CAS Registry Numbers for easy and accurate substance identification.

Searched by Barb O'Bryen, STIC 308-4291

L25 STR
L28 29 SEA FILE=REGISTRY SSS FUL L25
L30 12 SEA FILE=USPATFULL ABB=ON L28

previously printed

L34 NOT L29

L35 37 SEA FILE=HCAPLUS ABB=ON L28

FILE 'HCAPLUS' ENTERED AT 12:35:47 ON 09 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)

FILE 'USPATFULL' ENTERED AT 12:35:47 ON 09 APR 2003
CA INDEXING COPYRIGHT (C) 2003 AMERICAN CHEMICAL SOCIETY (ACS)
PROCESSING COMPLETED FOR L34
PROCESSING COMPLETED FOR L35

L36 32 DUP REM L34 L35 (5 DUPLICATES REMOVED)
ANSWERS '1-25' FROM FILE HCAPLUS
ANSWERS '26-32' FROM FILE USPATFULL

D QUE ABB HIT L36 1-32 fil cao; d que nos 132

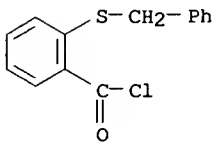
L36 ANSWER 1 OF 32 HCAPLUS COPYRIGHT 2003 ACS DUPLICATE 1
ACCESSION NUMBER: 2002:814891 HCAPLUS
DOCUMENT NUMBER: 137:325335
TITLE: Preparation of (hetero)arylamides as inhibitors of microsomal triglyceride transfer protein
INVENTOR(S): Booth, Richard John; Lee, Helen Tsenwhei; Pontrello, Jason Keith; Ramharack, Randy Ranjee; Roth, Bruce David
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 27 pp., Cont.-in-part of U.S. Ser. No. 422,568.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2002156281	A1	20021024	US 2001-21633	20011212
PRIORITY APPLN. INFO.:			US 1998-107119P P	19981105
			US 1999-422568 B2	19991021

OTHER SOURCE(S): MARPAT 137:325335
AB R3(CH2)nNR1COR2 [I, R1 = (substituted) pyridyl, pyridylmethyl, Ph, quinolyl, benzothienyl, etc.; R2 = Ph, PhCH2OC6H4, PhCH2SC6H4, PhCH2SOC6H4, naphthylmethyl, benzodioxanyl, benzothienyl, amino, aminoalkyl, etc.; R3 = biphenyl, benzothienyl, tetramethyltetralinyl, naphthalenyl; n = 0-2], were prepd. Thus, reaction of 2-ethoxy-N-pyridin-3-ylbenzamide and 2-phenylbenzyl bromide gave N-biphenyl-2-ylmethyl-2-ethoxy-N-pyridin-3-ylbenzamide. The latter inhibited lipoprotein A3 prodn. with IC50 = 0.9 .mu.M. The present invention also provides pharmaceutical compns. comprising I and methods of treatment of atherosclerosis, obesity, restenosis, coronary heart disease, hyperlipoproteinemia, hypercholesterolemia, and hypertriglyceridemia.
IT **1531-81-3**
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. of (hetero)arylamides as inhibitors of microsomal triglyceride

Searched by Barb O'Bryen, STIC 308-4291

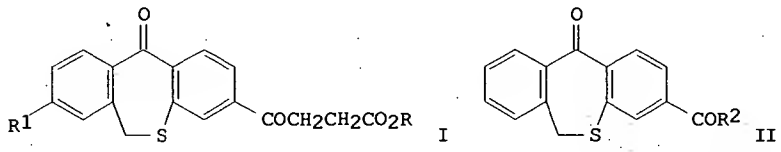
transfer protein)
RN 1531-81-3 HCAPLUS
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 2 OF 32 HCAPLUS COPYRIGHT 2003 ACS DUPLICATE 2
ACCESSION NUMBER: 1979:137704 HCAPLUS
DOCUMENT NUMBER: 90:137704
TITLE: 4-(8X-6,11-Dihydro-11-oxo-3-dibenzo[b,e]thiepinyl)-4-oxobutyric acids
INVENTOR(S): Ackrell, Jack
PATENT ASSIGNEE(S): Syntex (U.S.A.), Inc., USA
SOURCE: U.S., 13 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4130654	A	19781219	US 1978-873300	19780130
EP 3422	A1	19790808	EP 1979-300112	19790123
R: BE, CH, DE, FR, GB, LU, NL, SE				
JP 54117489	A2	19790912	JP 1979-6216	19790124
ES 477163	A1	19791016	ES 1979-477163	19790125

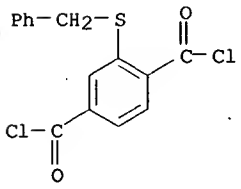
PRIORITY APPLN. INFO.: US 1978-873300 19780130
GI



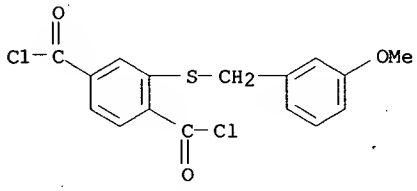
AB The title compds. (I, R = H, alkyl, cation; R1 = H, OMe, Cl) were prepd. Thus, nitroterephthalic acid was esterified, and treated with PhCH2SH, followed by hydrolysis to give benzylthioterephthalic acid, which was converted to acid chloride and subjected to intramol. Friedel-Crafts reaction to give II (R2 = Cl). Treatment of II (R2 = Cl) with CH2N2 gave II (R2 = CHN2), which was treated with HCl to give II (R2 = CH2Cl). Reaction of II (R2 = CH2Cl) with CH2(CO2Me)2 gave II [R2 = CH2CH(CO2Me)2], which on ester hydrolysis and decarboxylation gave I (R = R1 = H). I (R = R1 = H) had 27 times antiinflammatory activity of phenylbutazone.
IT **61220-65-3P 69646-81-7P 69646-82-8P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(prepn. and intramol. Friedel-Crafts reaction of)
RN 61220-65-3 HCAPLUS

Searched by Barb O'Bryen, STIC 308-4291

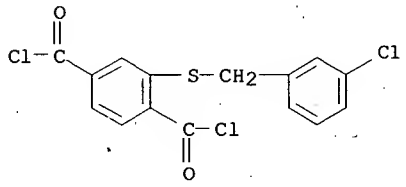
CN 1,4-Benzenedicarbonyl dichloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



RN 69646-81-7 HCAPLUS
CN 1,4-Benzenedicarbonyl dichloride, 2-[[(3-methoxyphenyl)methyl]thio]- (9CI) (CA INDEX NAME)



RN 69646-82-8 HCAPLUS
CN 1,4-Benzenedicarbonyl dichloride, 2-[[(3-chlorophenyl)methyl]thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 3 OF 32 HCAPLUS COPYRIGHT 2003 ACS DUPLICATE 3
ACCESSION NUMBER: 1978:22671 HCAPLUS
DOCUMENT NUMBER: 88:22671
TITLE: 6,11-Dihydrodibenzo[b,e]thiepin-11-one-3-carboxaldehyde
INVENTOR(S): Prince, Anthony; Halpern, Otto; Ackrell, Jack
PATENT ASSIGNEE(S): Syntex (U.S.A.), Inc., USA
SOURCE: U.S., 4 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

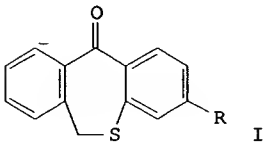
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4051148	A	19770927	US 1976-697648	19760618
SE 7608718	A	19771219	SE 1976-8718	19760803

Searched by Barb O'Bryen, STIC 308-4291

FI 7602234	A	19771219	FI 1976-2234	19760804
DK 7603510	A	19771219	DK 1976-3510	19760804
NO 7602722	A	19771220	NO 1976-2722	19760805
ES 459896	A1	19780816	ES 1977-459896	19770617
DK 7800238	A	19780117	DK 1978-238	19780117

PRIORITY APPLN. INFO.: US 1976-697648 19760618
DK 1976-3510 19760804

GI



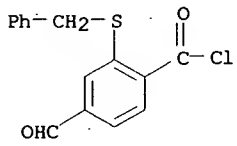
I

AB Cyclization of 4,3-(ClCO)(PhCH2S)C6H3CHO by anhyd. AlCl3 gave aldehyde I (R = CHO), which was treated with NaOMe and ClCH2CN to give I (R = 3-cyano-2-oxiranyl) (II). Cleavage of II with HBr, followed by treatment with Ac2O-pyridine gave I [R = CHBrCH(OAc)CN], which was dehydrobrominated to give I [R = CH:C(CN)OAc] (III). Acid or basic hydrolysis of III gave I (R = CH2CO2H).

IT **64976-84-7**
RL: RCT (Reactant); RACT (Reactant or reagent)
(cyclization of)

RN 64976-84-7 HCAPLUS

CN Benzoyl chloride, 4-formyl-2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

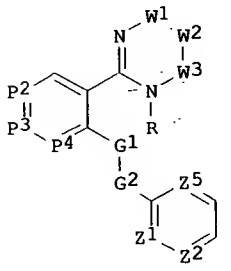


L36 ANSWER 4 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 2001:115125 HCAPLUS
DOCUMENT NUMBER: 134:178566
TITLE: Preparation of melanocortin-4 receptor binding compounds
INVENTOR(S): Maguire, Martin P.; Dai, Mingshi; Vos, Tricia J.
PATENT ASSIGNEE(S): Millennium Pharmaceuticals, Inc., USA
SOURCE: PCT Int. Appl., 215 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

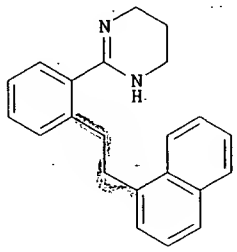
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001010842	A2	20010215	WO 2000-US21327	20000804
WO 2001010842	A3	20010816		

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT,

LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU,
SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN,
YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY,
DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ,
CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
EP 1204645 A2 20020515 EP 2000-953837 20000804
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
IE, SI, LT, LV, FI, RO, MK, CY, AL
BR 2000012984 A 20020716 BR 2000-12984 20000804
PRIORITY APPLN. INFO.: US 1999-147288P P 19990804
US 2000-223277P P 20000803
WO 2000-US21327 W 20000804
OTHER SOURCE(S): MARPAT 134:178566
GI



I

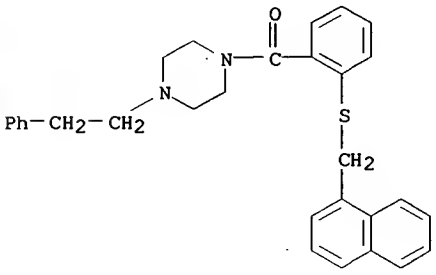


II

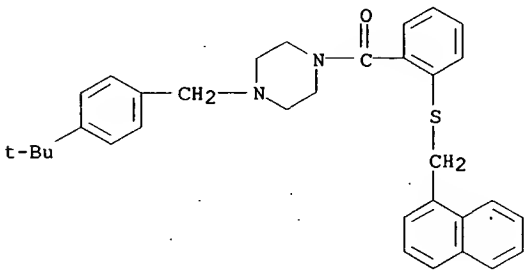
AB The title compds. of formula B-Z-E [wherein B = an anchor moiety; Z = a central moiety; E = an MC4-R interacting moiety], e.g. I [wherein P2, P3, and P4 = independently CH, CF, CCl, CBr, C(alkyl), C(alkoxy), C(CN), C(OH), or CI; W1 = covalent bond or CH2; W2 = CH2, CHR3, or CR3R4; W3 = CH2, CHR5, or CR5R6; R = H or alkyl; Z1 = CH or covalently linked to Z2 to form a naphthyl ring; Z2 = CH, C(C.tplbond.CH), CCl, CBr, CI, CF, or covalently linked to Z1 to form a naphthyl ring; Z5 = CH or C(OMe); R3-R6 = independently Me or Et], were prepd. and tested as melanocortin-4 receptor (MC4-R) binding agonists and antagonists. For example, .alpha.-tolunitrile in THF was added to a soln. of diisopropylamine in THF, which had been cooled to -78.degree.C and treated with BuLi. HMPA and 1-chloromethylnaphthalene in THF were added, the reaction cooled and stirred for 1 h, and the reaction quenched with H2O to give 2-(2-naphthalen-1-ylethyl)benzonitrile. Treatment with H2S and 1,3-diaminopropane, followed by heating to 80.degree.C for 72 h and work up, gave II. In a scincillation proximity assay (SPA) using high-throughput receptor binding screening, II showed exemplary inhibition of MC4-R. The invention compds., primarily 2-(2-arylalkylsulfanylphenyl)-4,5-dihydro-1H-imidazole and 1,4,5,6-tetrahydropyrimidine derivs., are useful in the treatment of disorders assocd. with wt. loss and pigmentation (no data).

IT **326485-07-8P 326485-08-9P 326485-37-4P**
326485-75-0P
RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation)
(inactive as MC4-R binding compd.; prepn. and high throughput MC4-R receptor binding screening of arylalkylsulfanylphenyl-substituted imidazoles and pyrimidines and analogs)

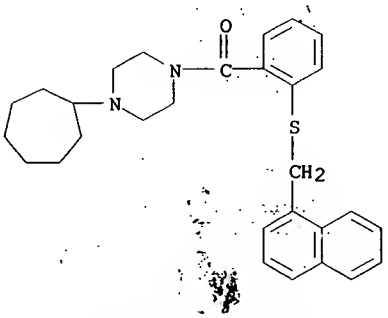
RN 326485-07-8 HCAPLUS
CN Piperazine, 1-[2-[(1-naphthalenylmethyl)thio]benzoyl]-4-(2-phenylethyl)- (9CI) (CA INDEX NAME)



RN 326485-08-9 HCAPLUS
CN Piperazine, 1-[[4-(1,1-dimethylethyl)phenyl]methyl]-4-[2-[(1-naphthalenylmethyl)thio]benzoyl]- (9CI) (CA INDEX NAME)

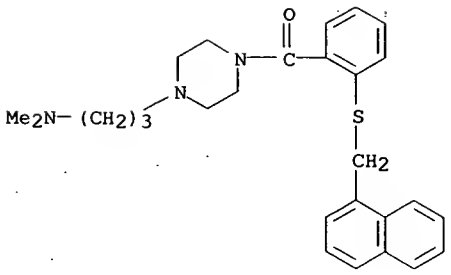


RN 326485-37-4 HCAPLUS
CN Piperazine, 1-cycloheptyl-4-[2-[(1-naphthalenylmethyl)thio]benzoyl]- (9CI) (CA INDEX NAME)

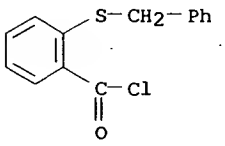


RN 326485-75-0 HCAPLUS
CN 1-Piperazinepropanamine, N,N-dimethyl-4-[2-[(1-naphthalenylmethyl)thio]benzoyl]- (9CI) (CA INDEX NAME)

Searched by Barb O'Bryen, STIC 308-4291



L36 ANSWER 5 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 2001:166492 HCAPLUS
DOCUMENT NUMBER: 134:326427
TITLE: A novel synthesis of [1]benzothieno[3,2-b][1]benzofuran
AUTHOR(S): Cernovska, Katerina; Nic, Miloslav; Pihera, Pavel; Svoboda, Jiri
CORPORATE SOURCE: Department of Organic Chemistry, Institute of Chemical Technology, Prague, Prague, 16628/6, Czech Rep.
SOURCE: Collection of Czechoslovak Chemical Communications (2000), 65(12), 1939-1949
CODEN: CCCCAK; ISSN: 0010-0765
PUBLISHER: Institute of Organic Chemistry and Biochemistry, Academy of Sciences of the Czech Republic
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:326427
AB A new synthesis of the title compd. based on the formation of the furan ring in the key step was elaborated. Me 2-methoxy[1]benzothieno[3,2-b][1]benzofuran-7-carboxylate was prepd. by this methodol. as a new type of a core for liq. crystal synthesis.
IT **1531-81-3**
RL: RCT (Reactant); RACT (Reactant or reagent)
(acylation of phenolic phosphonium bromide salt with acid chlorides)
RN 1531-81-3 HCAPLUS
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



bad data

REFERENCE COUNT: 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L36 ANSWER 6 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1996:543966 HCAPLUS
DOCUMENT NUMBER: 125:184898
TITLE: Structure-Activity Relationships of a Series of Novel (Piperazinylbutyl)thiazolidinone Antipsychotic Agents Related to 3-[4-[4-(6-Fluorobenzo[b]thien-3-yl)-1-piperazinyl]butyl]-2,5,5-trimethyl-4-thiazolidinone Maleate

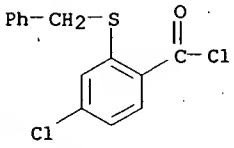
Searched by Barb O'Bryen, STIC 308-4291

AUTHOR(S): Hibb, Nicholas J.; Jurcak, John G.; Bregna, Deborah E.; Burgher, Kendra L.; Hartman, Harold B.; Kafka, Sharon; Kerman, Lisa L.; Kongsamut, Sam; Roehr, Joachim E.; et al.
CORPORATE SOURCE: Hoechst Marion Roussel Inc, Bridgewater, NJ, 08876, USA
SOURCE: Journal of Medicinal Chemistry (1996), 39(20), 4044-4057
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

AB HP-236 (3-[4-[4-(6-fluorobenzo[b]thien-3-yl)-1-piperazinyl]butyl]-2,5,5-trimethyl-4-thiazolidinone maleate) displayed a pharmacol. profile indicative of potential atypical antipsychotic activity. A series of piperazinylbutylthiazolidinones structurally related to this compd. were prepd. and evaluated in vitro for dopamine D2 and serotonin 5HT2 and 5HT1A receptor affinity. The compds. were examd. in vivo in animal models of potential antipsychotic activity and screened in models predictive of extrapyramidal side effect (EPS) liability. The synthesis of these compds., details of their structure-activity relationships, and discovery of a new lead, compd. are described.

IT **40183-55-9P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(structure-activity relations of (piperazinylbutyl)thiazolidinone antipsychotics)

RN 40183-55-9 HCAPLUS
CN Benzoyl chloride, 4-chloro-2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 7 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1993:517742 HCAPLUS
DOCUMENT NUMBER: 119:117742
TITLE: Organic nitrates, methods for preparing same, and use thereof for treating cardiovascular diseases
INVENTOR(S): Nallet, Jean Pierre; Dreux, Jacques; Berdeaux, Alain; Richard, Vincent; Martorana, Piero; Bohn, Helmut
PATENT ASSIGNEE(S): Laboratoires Hoechst, Fr.
SOURCE: PCT Int. Appl., 96 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

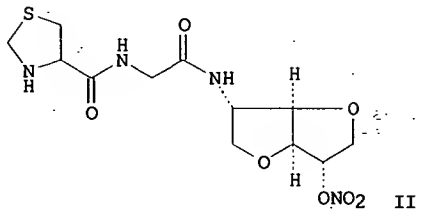
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9303037	A1	19930218	WO 1992-EP1746	19920801
W: CA, HU, JP, KR, US				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE				
FR 2680173	A1	19930212	FR 1991-10039	19910807
FR 2680173	B1	19950505		

Searched by Barb O'Bryen, STIC 308-4291

CA 2113922	AA	19930218	CA 1992-2113922	19920801
EP 530887	A1	19930310	EP 1992-202500	19920801
R: PT				
EP 604459	A1	19940706	EP 1992-917213	19920801
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, SE				
JP 07500817	T2	19950126	JP 1992-503265	19920801
HU 70546	A2	19951030	HU 1994-327	19920801
US 5591758	A	19970107	US 1993-971812	19930504
PRIORITY APPLN. INFO.:			FR 1991-10039	19910807
			WO 1992-EP1746	19920801

OTHER SOURCE(S): MARPAT 119:117742

GI



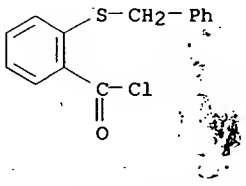
II

AB Org. nitrates RCOAnYB [I; R = many possible groups, particularly S-contg. residues, including thiazolidines and S-contg. amino acids; A = particularly CH2 or a substituted amino acid; n = 0, 1, >1; Y = O, NH; B = particularly dianhydro-1,4:3,6-hexitol mononitrate residues, itol nitrate residues, inositol nitrate residues] were prepd. as vasorelaxants for treatment of cardiovascular diseases, particularly angina pectoris, and show diminished tachyphylaxis. For example, amidation of 1,4:3,6-dianhydro-5-deoxy-5-amino-L-iditol 2-nitrate with N-(tert-butoxycarbonyl)glycine (72%), followed by deprotection with HCl-MeOH (85%), neutralization of the HCl salt (90%), a 2nd amidation with N-(tert-butoxycarbonyl)-L-thioproline using DCC (71%), and deprotection with HCl-EtOAc (76%), gave title compd. L-II as the HCl salt (III). Prepns. of over 55 I and 17 precursors, and detailed results of a variety of hemodynamic tests on several I are given. In comparison with isosorbide mononitrate, III showed higher potency, longer duration of action, and an absence of tachyphylaxis.

IT **1531-81-3**, S-Benzylthiosalicylic acid chloride
RL: RCT (Reactant); RACT (Reactant or reagent)
(esterification and amidation of, in prepn. of vasorelaxants)

RN 1531-81-3 HCAPLUS

CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 8 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1991:655826 HCAPLUS
DOCUMENT NUMBER: 115:255826
TITLE: Preparation of propanediamine derivatives as ligands

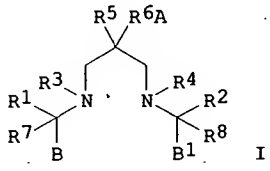
Searched by Barb O'Bryen, STIC 308-4291

for radioactive isotopes, their metal complexes, and their use in diagnosis and therapy

INVENTOR(S): Neumeier, Reinhard; Kramp, Wolfgang; Maecke, Helmut R.
PATENT ASSIGNEE(S): Institut fuer Diagnostikforschung G.m.b.H., Germany
SOURCE: Eur. Pat. Appl., 29 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 417870	A2	19910320	EP 1990-250214	19900820
EP 417870	A3	19910626		
EP 417870	B1	19940720		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE				
DE 3930674	A1	19910321	DE 1989-3930674	19890911
NO 9003551	A	19910312	NO 1990-3551	19900813
NO 173234	B	19930809		
NO 173234	C	19931117		
HU 59370	A2	19920528	HU 1990-5026	19900815
CA 2023595	AA	19910312	CA 1990-2023595	19900820
ES 2060002	T3	19941116	ES 1990-250214	19900820
ZA 9006634	A	19910626	ZA 1990-6634	19900821
US 5302370	A	19940412	US 1990-572140	19900822
AU 9061290	A1	19910314	AU 1990-61290	19900823
AU 641421	B2	19930923		
IL 95547	A1	19960514	IL 1990-95547	19900831
DD 297636	A5	19920116	DD 1990-343845	19900905
JP 03188048	A2	19910816	JP 1990-239148	19900911

PRIORITY APPLN. INFO.: DE 1989-3930674 19890911
OTHER SOURCE(S): MARPAT 115:255826
GI



I

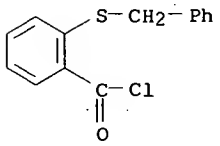
AB The title ligands [I; R1, R2, R5 = H, (HO-substituted) C1-6 alkyl; R3, R4 = H, (amino)C1-6 alkyl, HO2CCH2, (C1-6 alkoxycarbonyl)methyl or -benzyl; R6 = C1-6 alkylene; R7, R8 = H, C1-6 alkyl; B, B1 = Ph, 2-HSC6H4, naphthyl, thienyl, pyrrolyl, all optionally substituted by 1-3 HO), CH(NO)R9; R9 = C1-6 alkyl; R1R9, R2R9 can form a 5- or 6-membered ring with (CH2)3 or (CH2)4; A = functional group Z, a compd. T bound to R6 via Z and capable of accumulating itself in lesions or specific tissues, e.g. an enzyme, amino acid, saccharide, a growth factor, esp. a monoclonal antibody or its fragments, biotin, and misonidazole; Z = amino, carboxy, HO, oxiranyl, aminophenyl, C2-6 alkenyl, etc.], useful in tumor diagnosis and therapy, were prepd. Condensation of 4-O2NC6H4CH(CH2NH2)2 [prepn. from CH2(CO2Et)2 and 4-O2NC6H4CH2Br given] with 2-chloro-2-methyl-3-nitrosobutane gave 27% 6-(4'-nitrobenzyl)-3,3,9,9-tetramethyl-4,8-diazaundecane-2,10-dione dioxime. This was reduced (26%) to its 4'-aminobenzyl analog, chelated by Cu(OAc)2 (45%), the Cu-chelate coupled (75%) at position 4' with biotin N-hydroxysuccinimide ester, the resulting biotin conjugate decomplexed (41%) by KCN, and the ligand recomplexed with

a radioactive tracer: technetium-99m (200 .mu.Ci). A rat left hind leg muscle was injected with 20 .mu.L of a com. streptavidin-Sepharose conjugate and, 30 min later, with 5 .mu.g (i.v.) of the latter chelate (purity >90%). After 4 h, the radioactivity in the left hind leg was 14-fold higher than in the right hind leg, and it contained 1.4% of the total of the applied dosis/g muscle.

IT **1531-81-3**, S-Benzylthiosalicylic acid chloride
RL: RCT (Reactant); RACT (Reactant or reagent)
(acylation by, of propanediamine deriv., in prepn. of bidentate ligands)

RN 1531-81-3 HCAPLUS

CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 9 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1989:207841 HCAPLUS
DOCUMENT NUMBER: 110:207841
TITLE: Herbicidal sulfonamides
INVENTOR(S): Rorer, Morris Padgett
PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA
SOURCE: Eur. Pat. Appl., 276 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 301784	A1	19890201	EP 1988-306806	19880725
R: ES, GR				
US 4906282	A	19900306	US 1988-204556	19880615
WO 8900991	A1	19890209	WO 1988-US2459	19880725
W: AU, JP				
RW: AT, BE, CH, DE, FR, GB, IT, LU, NL, SE				
AU 8821334	A1	19890301	AU 1988-21334	19880725
AU 611191	B2	19910606		
EP 386001	A1	19900912	EP 1988-906577	19880725
R: AT, BE, CH, DE, FR, GB, IT, LI, LU, NL, SE				
JP 02504275	T2	19901206	JP 1988-506452	19880725
US 4995901	A	19910226	US 1990-461581	19900105

PRIORITY APPLN. INFO.: US 1987-78191 19870727
US 1988-204556 19880615
WO 1988-US2459 19880725

OTHER SOURCE(S): MARPAT 110:207841

AB The sulfonamides JSO2NHC(:W)NRA (I) [J = (un)substituted Ph, naphthyl, thienyl, pyridinyl, pyrazolyl, etc.; W = O, S; R = H, Me; A = (un)substituted 1,2,4-triazolyl, pyrimidinyl, 1,3,5-triazinyl, etc.] are prepd. as herbicides. 2-[Cyano(methoxyimino)methyl]benzenesulfonamide (prepn. given) was reacted with Ph (4,6-dimethoxy-1,3,5-triazin-2-yl)carbamate, in dry acetonitrile, in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene, to give I [J = 2-[MeON:C(CN)]C6H4, W = O, R = H, A = 4,6-dimethoxy-1,3,5-triazin-2-yl] (II). Pre-emergence application of 0.05 kg II/ha controlled velvet-leaf (Abutilon

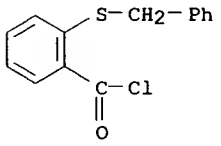
Searched by Barb O'Bryen, STIC 308-4291

theophrasti), morning-glory (Ipomoea) and other weeds. A wettable powder comprised I [J = 2-[MeON:C(CN)]C6H4, W = O, R = H, A = 4-methoxy-6-methyl-2-pyrimidinyl] 65, dodecylphenol polyethylene glycol ether 2, Na lignin sulfonate 4, Na silicoaluminate 6 and montmorillonite 23%.

IT **1531-81-3P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(prepn. and reaction of, with methoxylamine)

RN 1531-81-3 HCAPLUS

CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 10 OF 32 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1989:515023 HCAPLUS

DOCUMENT NUMBER: 111:115023

TITLE: Pyrrole derivatives as cardiotonics, process for their preparation and pharmaceutical compositions containing them

INVENTOR(S): Dixon, John; Baxter, Andrew John Gilby; Manners, Carol Nancy; Teague, Simon

PATENT ASSIGNEE(S): Fisons PLC, UK

SOURCE: Eur. Pat. Appl., 69 pp.
CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 300688	A1	19890125	EP 1988-306464	19880714
R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, LU, NL, SE				
DK 8804049	A	19890122	DK 1988-4049	19880720
JP 01061455	A2	19890308	JP 1988-179286	19880720

PRIORITY APPLN. INFO.: GB 1987-17193 19870721
GB 1987-30116 19871224

OTHER SOURCE(S): MARPAT 111:115023

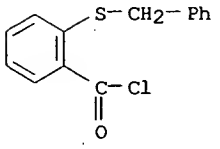
GI For diagram(s), see printed CA Issue.

AB Title compds. I [R1 = R11, NHR11, NHCO2R11 wherein R11 = H, C1-6 alkyl; R2, R5 = OH, halo, NO2, etc.; G = (CH2)zWy in which W = CO, SOq, etc.; q = 0-2; z = 0-3; y = 0 or 1 (or 2 provided W = CO); up to 2 of the methylene segments in the chain (CH2)z are optionally replaced by NH and one segment is optionally replaced by O, etc.; the chain is optionally unsatd. and optionally substituted by C1-6 alkyl, alkoxy, etc.; A = (substituted) 5- or 6-membered ring or a bicyclic or tricyclic fused ring system; R3 = H, NO2, CN, halo, etc.; several provisos are given], useful as cardiotonics (no data), were prepd. A mixt. of 2-((4-nitrophenyl)thio)benzoyl chloride, Me 2,5-dimethyl-1H-pyrrole-3-carboxylate, and AlCl3 in CH2Cl2 was stirred at room temp. for 16 h to give Me 2,5-dimethyl-4-(2-((4-nitrophenyl)thio)benzoyl)-1H-pyrrole-3-carboxylate.

IT **1531-81-3**
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, in prepn. of cardiotonic)

Searched by Barb O'Bryen, STIC 308-4291

RN 1531-81-3 HCAPLUS
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

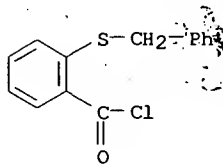


L36 ANSWER 11 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1989:646719 HCAPLUS
DOCUMENT NUMBER: 111:246719
TITLE: Molybdenum(VI)-dioxo complexes with linear and tripodal tetradentate ligands: models for the molybdenum(VI/V) centers of the molybdenum hydroxylases and related enzymes. 1. Syntheses and structures
AUTHOR(S): Hinshaw, Carol J.; Peng, Gang; Singh, Raghuvir; Spence, Jack T.; Enemark, John H.; Bruck, Michael; Kristofzski, John; Merbs, Shannath L.; Ortega, Richard B.; Wexler, Pamela A.
CORPORATE SOURCE: Dep. Chem. Biochem., Utah State Univ., Logan, UT, 84322-0300, USA
SOURCE: Inorganic Chemistry (1989), 28(25), 4483-91
CODEN: INOCAJ; ISSN: 0020-1669
DOCUMENT TYPE: Journal
LANGUAGE: English

AB As models for the molybdenum(VI/V) centers of the molybdenum hydroxylases and related enzymes, 15 new Mo(VI)-dioxo complexes (MoO_2L) with tetradentate ligands were prepd. and characterized. The effects of coordinating groups (N_2S_2, N_2OS, and N_2O_2), chelate ring size (five and six members), ligand geometry (linear and tripodal), and steric bulk were studied. X-ray crystal structures were obtained for seven of the complexes. While minor differences, attributed to these features, are evident, the structures have remarkably similar Mo-ligand bond lengths and bond angles and all have distorted-octahedral geometry. The oxo groups are cis to one another and to the thiolate or phenolate groups of the ligands. The N atoms are approx. trans to the oxo groups, and the Mo-N bonds are relatively long (>2.34 ANG.), with the bond length correlated with the size of the trans O=Mo-N bond angle. The Mo=O and M-S(thiolate) bond lengths are comparable to those detd. by EXAFS spectroscopy for the Mo centers of the enzymes. The relevance of the results to the structures of the Mo centers of the enzymes is discussed.

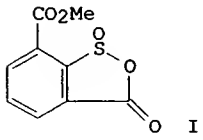
IT **1531-81-3P**, S-Benzylthiosalicylic acid chloride
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(prepn. and substitution reaction of, with dimethylethylenediamine)

RN 1531-81-3 HCAPLUS
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



Searched by Barb O'Bryen, STIC 308-4291

L36 ANSWER 12 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1980:181052 HCAPLUS
DOCUMENT NUMBER: 92:181052
TITLE: The first isolated sulfinyl carboxylate; crystal and molecular structure
AUTHOR(S): Walter, Wolfgang; Krische, Bernd; Adiwidjaja, Gunadi
CORPORATE SOURCE: Inst. Org. Chem. Biochem., Univ. Hamburg, Hamburg, D-2000/13, Fed. Rep. Ger.
SOURCE: Liebigs Annalen der Chemie (1980), (1), 14-27
CODEN: LACHDL; ISSN: 0170-2041
DOCUMENT TYPE: Journal
LANGUAGE: German
GI

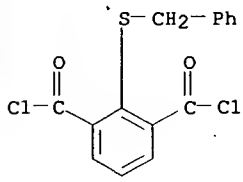


I

AB I and several other cyclic sulfinyl carboxylates were prepd. by s-oxidn. of the corresponding sulfenyl carboxylates using m-ClC6H4CO2OH. X-ray data showed that, with the exception of the exocyclic sulfinyl O, the I mol. is nearly planar and reveals only a small amt. of no-bond resonance compared with the sulfenyl carboxylate.

IT **67666-72-2**
RL: PRP (Properties)
(spectra of)

RN 67666-72-2 HCAPLUS
CN 1,3-Benzenedicarbonyl dichloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 13 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1980:163853 HCAPLUS
DOCUMENT NUMBER: 92:163853
TITLE: 6,11-Dihydrodibenzothiepin-11-ones and their S-oxides and pharmaceutical compositions containing them
INVENTOR(S): Ackrell, Jack
PATENT ASSIGNEE(S): Syntex (U.S.A.), Inc., USA
SOURCE: Eur. Pat. Appl., 54 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1

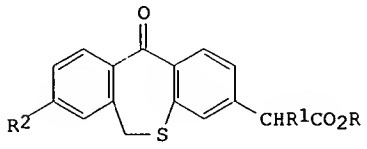
Searched by Barb O'Bryen, STIC 308-4291

PATENT INFORMATION:

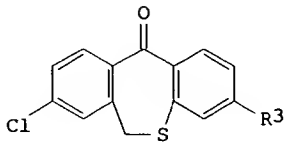
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 5055	A2	19791031	EP 1979-300645	19790419
R: BE, CH, DE, FR, GB, NL, SE				
AU 7946181	A1	19791025	AU 1979-46181	19790418
DK 7901628	A	19791022	DK 1979-1628	19790420
JP 54141793	A2	19791105	JP 1979-48568	19790421

PRIORITY APPLN. INFO.: US 1978-898602 19780421

GI



I

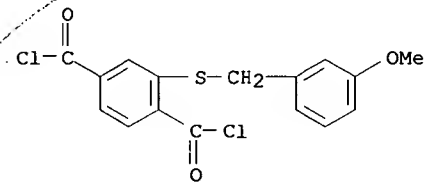


II

AB Dibenzothiepinalkanoic acids I (R = H, alkyl; R1 = H, Me; R2 = H, Cl, OMe) and their S-oxides were prepd. Thus, nitroterephthalic acid was esterified and treated with 3-ClC6H4CH2SH to give 2,5-(Me2CHO2C)2C6H3SCH2C6H4Cl-3, which was hydrolyzed to the acid and chlorinated. The resulting 2,5-(ClCO)2C6H3SCH2C6H4Cl-3 was cyclized with AlCl3 to give II (R3 = COCl), which was treated with CH2N2 to give II (R3 = COCHN2). The latter compd. was rearranged and methanolyzed to give II (R3 = CH2CO2Me). Ester hydrolysis gave II (R3 = CH2CO2H) which at 0.4 mg topically decreased the wt. of edematous skin disks from 500 to 147.2 mg.

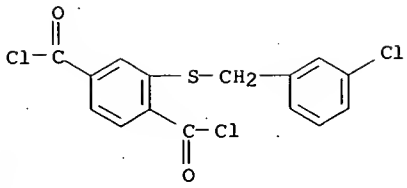
IT **69646-81-7P 69646-82-8P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(prepn. and cyclization of)

RN 69646-81-7 HCAPLUS
CN 1,4-Benzenedicarbonyl dichloride, 2-[[(3-methoxyphenyl)methyl]thio]- (9CI) (CA INDEX NAME)



RN 69646-82-8 HCAPLUS
CN 1,4-Benzenedicarbonyl dichloride, 2-[[(3-chlorophenyl)methyl]thio]- (9CI) (CA INDEX NAME)

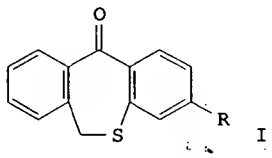
Searched by Barb O'Bryen, STIC 308-4291



L36 ANSWER 14 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1978:105182 HCAPLUS
DOCUMENT NUMBER: 88:105182
TITLE: 11-Oxo-6,11-dihydrodibenzo[b,e]thiepin-3-acetaldehydes and 3-acetals
INVENTOR(S): Ackrell, Jack
PATENT ASSIGNEE(S): Syntex (U.S.A.), Inc., USA
SOURCE: Ger. Offen., 51 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2729120	A1	19780112	DE 1977-2729120	19770628
US 4066663	A	19780103	US 1976-701780	19760701
BE 856144	A1	19771227	BE 1977-178807	19770627
NL 7707136	A	19780103	NL 1977-7136	19770628
GB 1532205	A	19781115	GB 1977-27053	19770628
ZA 7703933	A	19790228	ZA 1977-3933	19770629
JP 53005182	A2	19780118	JP 1977-78462	19770630
FR 2356647	A1	19780127	FR 1977-20211	19770630
FR 2356647	B1	19790720		
ES 460297	A1	19780816	ES 1977-460297	19770630
AU 7726605	A1	19790104	AU 1977-26605	19770630
ES 469913	A1	19781216	ES 1978-469913	19780516

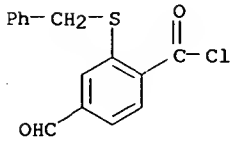
PRIORITY APPLN. INFO.: US 1976-701780 19760701
GI



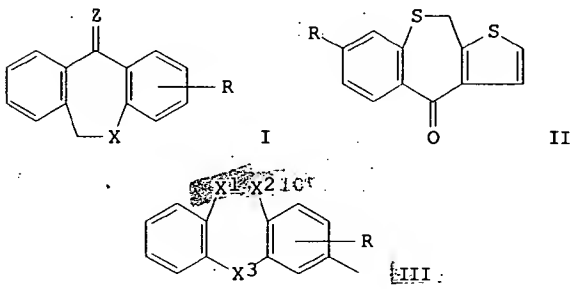
AB The title compds. I [R = CHR1CHO, CHR1CH(OR2)OR3; R1 = H, Me; R2 = R3 = C1-6 alkyl] were prepd. for use as analgesics, antipyretics, and antiinflammatory agents at 0.5-15mg/kg. Thus, I (R = CHO) reacted with MeOCH2P+Ph3Cl- to give I (R = CH:CHOMe), which was treated with MeOH in the presence of HClO4 to give I [R = CH2CH(OMe)2].
IT **64976-84-7**
RL: RCT (Reactant); RACT (Reactant or reagent)
(prepn. and cyclization of)
RN 64976-84-7 HCAPLUS

Searched by Barb O'Bryen, STIC 308-4291

CN Benzoyl chloride, 4-formyl-2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 15 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1978:557159 HCAPLUS
DOCUMENT NUMBER: 89:157159
TITLE: Synthesis and antiinflammatory activity of 6,11-dihydro-11-oxodibenzo[b,e]thiepinalkanoic acids and related compounds
AUTHOR(S): Ackrell, Jack; Antonio, Yulia; Franco, Fidencio; Landeros, Rosita; Leon, Alicia; Muchowski, Joseph M.; Maddox, Michael L.; Nelson, Peter H.; Rooks, Wendell H.; et al.
CORPORATE SOURCE: Res. Lab., Syntex, S. A., Mexico City, Mex.
SOURCE: Journal of Medicinal Chemistry (1978), 21(10), 1035-44
CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English
GI

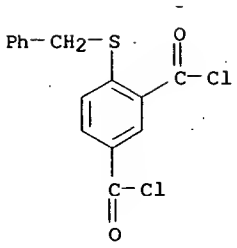


AB The title compds. I-III [X = S, SO2; X1 = CO, CH2, CH(OH); X2 = S, CH2; X3 = S, SO2, O; or X1X2 = CH:CH; Z = O, H2, or H,OH; R = CHR1COR2 (R1 = H, Me, Et; R2 = OMe, OH) were prepd. and assayed for antiinflammatory activity. Also prepd. were I and II (R = COCl), which were transformed via Arndt-Eistert synthesis to I and II (R = CHR1COR2). Tiopinac (I, R = 3-CH2CO2H, X = S, Z = O) [61220-69-7] was prepd. and had a high antiinflammatory activity in both short and long term animal assays and a low gastric irritation liability in rats and dogs.

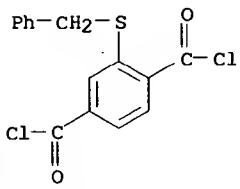
IT **67666-75-5P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(prepn. and hydrolysis and cyclization of)

RN 67666-75-5 HCAPLUS
CN 1,3-Benzenedicarbonyl dichloride, 4-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

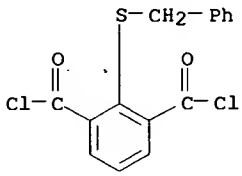
Searched by Barb O'Bryen, STIC 308-4291



IT **61220-65-3P 67666-72-2P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
RN 61220-65-3 HCAPLUS
CN 1,4-Benzenedicarbonyl dichloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



RN 67666-72-2 HCAPLUS
CN 1,3-Benzenedicarbonyl dichloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 16 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1977:5337 HCAPLUS
DOCUMENT NUMBER: 86:5337
TITLE: 6,11-Dihydrodibenzothiepin-11-ones
INVENTOR(S): Ackrell, Jack
PATENT ASSIGNEE(S): Syntex (U.S.A.), Inc., USA
SOURCE: Ger. Offen., 67 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

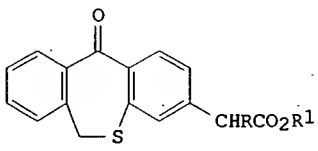
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

DE 2606312	A1	19760826	DE 1976-2606312	19760217
US 4000288	A	19761228	US 1975-634085	19751121
US 4000308	A	19761228	US 1975-634086	19751121
NL 7600899	A	19760820	NL 1976-899	19760129
NO 7600297	A	19760819	NO 1976-297	19760130
AU 7610686	A1	19770804	AU 1976-10686	19760130
AU 500501	B2	19790524		
FI 7600273	A	19760819	FI 1976-273	19760205
BE 838637	A1	19760817	BE 1976-164382	19760217
FR 2301245	A1	19760917	FR 1976-4353	19760217
FR 2301245	B1	19790720		
SE 7601754	A	19761025	SE 1976-1754	19760217
JP 51143688	A2	19761210	JP 1976-16446	19760217
PL 100687	P	19781031	PL 1976-197898	19760217
PL 100494	P	19781031	PL 1976-197897	19760217
SU 646910	D	19790205	SU 1976-2323954	19760217
DK 7600676	A	19760819	DK 1976-676	19760218
ES 445304	A1	19771001	ES 1976-445304	19760218
AT 352731	B	19791010	AT 1976-1159	19760218
AT 7601159	A	19790315		
SU 670223	D	19790625	SU 1977-2444050	19770126
SU 682131	D	19790825	SU 1977-2442950	19770126
SU 667135	D	19790605	SU 1977-2445098	19770128
ES 459400	A1	19780816	ES 1977-459400	19770601
ES 459401	A1	19790616	ES 1977-459401	19770601
DK 7800348	A	19780124	DK 1978-348	19780124
AT 352738	B	19791010	AT 1978-6087	19780821
AT 7806087	A	19790315		
AT 352739	B	19791010	AT 1978-6088	19780821
AT 7806088	A	19790315		
DK 7805102	A	19781116	DK 1978-5102	19781116
DK 7805101	A	19781116	DK 1978-5101	19781116
DK 7805100	A	19781116	DK 1978-5100	19781116

PRIORITY APPLN. INFO.:

US 1975-550316	19750218
US 1975-591725	19750630
US 1975-634085	19751121
US 1975-634086	19751121
DK 1976-676	19760218
AT 1976-1159	19780821

GI



I

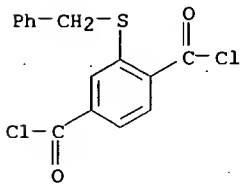
AB The title compds. (I; R = H, Me; R1 = H, Me, Et, PhCHMe, Me2CHCH2CH2, K, Ca, Cu), useful as inflammation inhibitors, are prepd. by cyclizatioon of (benzylthio)terephthalic acid derivs. Thus, cyclization of (benzylthio)terephthaloyl chloride in CH2Cl2 in presence of AlCl3 and MeNO2 5 hr at 25.degree. gives 70.7% 6,11-dihydro-11-oxo-dibenzo[b,e]thiepin-3-carbonyl chloride (II). Reaction of II with CH2N2 gives the 3-diazoacetyl analog (III). Treatment of 9.5 g III with PhCO2Ag 16 hr in refluxing MeOH gives 7 g I (R = H, R1 = Me).

IT **61220-65-3P**

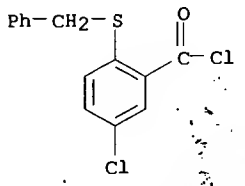
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

Searched by Barb O'Bryen, STIC 308-4291

(prepn. and cyclization of)
RN 61220-65-3 HCAPLUS
CN 1,4-Benzenedicarbonyl dichloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

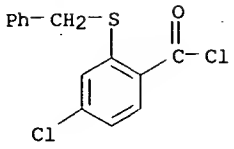


L36 ANSWER 17 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1973:84307 HCAPLUS
DOCUMENT NUMBER: 78:84307
TITLE: 1,2-Benzisothiazoles. IV. Preparation of the 3-methyl derivative from o-mercaptoacetophenone oxime
AUTHOR(S): Clarke, K.; Hughes, C. G.; Scrowston, R. M.
CORPORATE SOURCE: Dep. Chem., Univ. Hull, Hull, UK
SOURCE: Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999) (1973), (4), 356-9
CODEN: JCPRB4; ISSN: 0300-922X
DOCUMENT TYPE: Journal
LANGUAGE: English
GI For diagram(s), see printed CA Issue.
AB o-Mercaptoacetophenone oxime (I) with polyphosphoric acid gave a mixt. of 2-methylbenzothiazole (II), resulting from a Beckmann rearrangement prior to cyclization, and 3-methyl-1,2-benzoisothiazole (III). Similarly 4'-chloro-, 5'-chloro-, and 5'-nitro-2'-mercaptoacetophenone oxime gave mainly the corresponding benzothiazole. Cyclization of o-thiocyanatoacetophenone to give only III (Ricci, A.; Martani, A., 1963) was due to the initial formation of 2-imino-5-methyl-3,1,4-benzoxathiazepine rather than I.
IT **40183-37-7 40183-55-9**
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with diethyl ethoxymagnesiomalonate)
RN 40183-37-7 HCAPLUS
CN Benzoyl chloride, 5-chloro-2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



RN 40183-55-9 HCAPLUS
CN Benzoyl chloride, 4-chloro-2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

Searched by Barb O'Bryen, STIC 308-4291



L36 ANSWER 18 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1969:512799 HCAPLUS
DOCUMENT NUMBER: 71:112799
TITLE: Antiinflammatory 2-arylbenzo[b]thiophen-3(2H)-one 1,1-dioxides and 2-arylnaphtho[2,3-b]thiophen-3(2H)-one 1,1-dioxides
INVENTOR(S): Lombardino, Joseph G.
PATENT ASSIGNEE(S): Pfizer, Chas., and Co., Inc.
SOURCE: S. African, 43 pp.
CODEN: SFXXAB
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
ZA 6802803		19690102		
PRIORITY APPLN. INFO.:			US	19670502

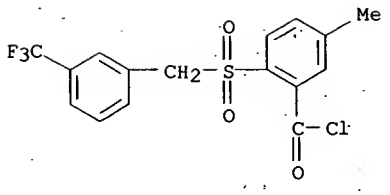
GI For diagram(s), see printed CA Issue.
AB Antiinflammatory compds., I and II, are prepd. Thus, to 85 ml. boiling H2O contg. 86 g. Na2S.9H2O and 11.2 g. powd. S was added 13 g. NaOH in 33 ml. H2O, the soln. cooled to 0.degree. and set aside (Soln. 1). 5-Methylanthranilic acid (50 g.) was added to a mixt. of 165 ml. H2O and 66 ml. concd. HCl, cooled to 0.degree. and treated with 23 g. NaNO2 in 93 ml. H2O over 10 min. at <5.degree. followed by addn. of 200 g. ice (Soln. 2). Soln. 2 was added to Soln. 1 at 0.degree. over 20-30 min., warmed to room temp., stirred 2 hrs. and acidified (HCl, Congo red) to give 66 g. bis(2-carboxy-4-tolyl disulfide (III). A mixt. of III so obtained and 45 g. Zn dust in 500 ml. AcOH was refluxed 4 hrs. and cooled to give 23 g. 5-(methylthio)salicyclic acid (IV), m. 163-4.degree.. 3-Mercapto-2-naphthoic acid, m. 219-21.degree., was similarly prepd. A soln. of 4.15 g. K2CO3 in 50 ml. H2O was treated with 100 ml. EtOH, 5.05 g. IV, 3.8 g. PhCH2Cl; after CO2 evolution stopped, the mixt. refluxed 1 hr., concd. in vacuo, dild. with 600 ml. H2O, filtered, and acidified yielded 6.9 g. 2,5-(PhCH2S)MeC6H3CO2H (V), m. 169-71.degree.. Similarly prepd. were 2,5-(3-O2NC6H4CH2S)-MeC6H3CO2H, m. 164-7.degree., 2,5-(3-CF3C6H4CH2S)MeC6H3CO2H, m. 153-5.degree., 2,5-(4-ClC6H4CH2S)MeC6H3CO2H, m. 188-91.degree., 3-[m-trifluoromethyl) benzylthio]-2-naphthoic acid, m. 222-5.degree., 3-benzylthio-2-naphthoic acid, m. 224-32.degree., and 3-(p-chlorobenzylthio)-2-naphthoic acid, m. 218-21.degree.. V (6.1 g.) was added to 200 ml. 97% HCO2H, heated at 54.degree., treated with 15 ml. 30% H2O2 25 min., heated 3 hrs. at 54.degree., after keeping at room temp. overnight concd. in vacuo to remove HCO2H, dried over P2O5 2 hrs. and triturated with 300 ml. H2O to give 6.5 g. 2,5-(benzylsulfonyl)-5-methylbenzoic acid (VI), m. 198-201.degree.. Similarly prepd. were 2,5-(3-O2NC6H4CH2SO2)MeC6H3CO2H, m. 213-16.degree., 2,5-(3-CF3-C6H4CH2SO2)MeC6H3CO2H (VII), m. 156-9.degree., 2,5-(p-ClC6H4-CH2SO2)MeC6H3CO2H, m. 184-6.degree., 3-(benzylsulfonyl)-2-naphthoic acid, m. 143-51.degree., 3-(p-chlorobenzylsul fonyl)-2-naphthoic acid, m. 207-9.degree., 3-[m-(trifluoromethyl)benzylsulfonyl]- 2-naphthoic acid, m.

Searched by Barb O'Bryen, STIC 308-4291

181-93.degree.. VI (5.7 g.) in 300 ml. alc. HCl was refluxed 15 hrs., left to stand at room temp. 2 days, concd. in vacuo and dild. with a mixt. of 400 ml. 10% NaHCO3 and ether. The aq. layer was extd. with 200 ml. ether, and the combined ether soln. was washed with H2O and concd. to give 5.8 g. 2,5-(PhCH2SO2)MeC6H3CO2Me (VIII). VIII in 200 ml. EtOH was treated with 80 ml. M NaOEt soln. in EtOH, refluxed for 1.5 hrs., concd. in vacuo, dild. with 250 ml. H2O and acidified (6N HCl) to give 3.75 g. I (R = Me, R1 = H), m. 181.5.degree.. Similarly prepd. were I (R = Me, R1 = 3-NO2), m. 212-14.degree., I (R = Me, R1 = 3-CF3) (IX), I (R = Me, R1 = 4-Cl). A mixt. of 8 g. VII and 50 ml. SOCl2 in 50 ml. dry C6H6 was refluxed 1 hr. under N atm., concd. in vacuo, treated with 50 ml. MeOH, refluxed for 1 hr. and concd. to give a solid product. The product, 2,5-(m-CF3C6H4CH2SO2)MeC6H3CO2Me, was cyclized in the previous manner to give IX. m, 142-5.degree.. Similarly prepd. were II (R = H), m. 170-3.degree., II (R = p-Cl), m. 235-7.degree., and II (R = m-CF3) (X), m. 188-9.degree.. Other I are also similarly prepd. 3-Amino-2-naphthoic acid (314 g.) in 2.5 l. H2O and 4.2 l. tetrahydrofuran was treated with 840 ml. concd. H2SO4 at <28.degree., cooled, treated with 137 g. NaNO2 in 2 l. H2O at <5.degree. over 45 min., stirred at -2.degree. for 15 min. and treated with 1.5 lb. (10.6 moles) SO2 over 5 min. at 0.degree. followed by addn. of 420 g. powd. Cu over 1.5 hrs. SO2 was passed into the mixt. 1 hr. (total amt. 3 lb.). The mixt. was warmed to 10.degree. slowly and, after 16 hrs. at room temp., the org. layer was filtered through C, concd. to 1.5 l., dild. with 5.5 l. CHCl3, concd. in vacuo to 2 l. and cooled to 18.degree. to give 200 g. 3-sulfino-2-naphthoic acid (XI), m. 142.3.degree.. A soln. of 118 g. XI, 102 g. Et3N, and 194.6 g. m-CF3C6H4Cl in 1 l. dry MeCN was refluxed 16 hrs., cooled to 8.degree., filtered from the HCl salt formed, concd. in vacuo, dild. with 600 ml. 5% HCl and extd. with ether to give 138 g. m-(trifluoromethyl)benzyl 3-[m-(trifluoromethyl)-benzylsulfonyl]-2-naphthoate (XII), m. 111-13.degree.. Similar cyclization of 111 g. XII with NaOMe gave 36 g. X. A mixt. of 0.5 mole PhCH2Cl and 0.5 mole thiourea in 250-400 ml. abs. EtOH was refluxed 3 hrs., treated with 300 ml. 10% NaOH soln., refluxed 2 hrs., concd. in vacuo, cooled, acidified and extd. with ether to give PhCH2SH (XIII). XIII (12.4 g.) in 100 ml. EtOH was treated with 100 ml. M NaOEt in EtOH under N atm., concd., dild. with 100 ml. dry Me2NCHO, treated with 21 g. 4,3-Cl(NC)C6H3CF3, and stirred 0.5 hr. to give 27.4 g. 2,5-(Ph-CH2S)F3CC6H3CN (XIV). A mixt. of 17.5 g. XIV in 15 ml. EtOH and 200 ml. 20% NaOH was refluxed 27 hrs., concd., extd. with ether (200 ml./3 times), concd., dild. with water and acidified (6N HCl) to give 15.7 g. 2,5-(PhCH2S)F3CC6H3CO2H (XV), m. 169-74.degree.. Similarly prepd. was 2,5-(m-MeC6H4CH2S)-F3CC6H3CO2H(XVI), m. 192-5.degree.. Oxidn. of XV and XVI gave 73% 2,5-(PhCH2SO2)F3CC6H3CO2H (XVII), m. 171-2.5.degree., and 80% 2,5-(m-MeC6H4CH2SO2)F3CC6H3CO2H (XVIII), m. 165-6.degree.. Cyclization of XVII and XVIII via esterification of the acid chlorides gave 83% I (R = CF3, R1 = H), m. 198-200.degree., and 90% I (R = CF3, R1 = m-Me), m. 174-6.degree.. 2,5-(m-MeC6H4CHS)O2N-C6H3CO2H, m. 238-40.degree., 2,5-(m-MeC6H4CH2SO2)O2NC6H3CO2H, m. 244-6.degree., and I (R = NO2, R1 = m- NO2), m. 137-40.degree., were similarly prepd. from m-MeC6H4CH2SH and 2,5-Cl(O2N)C6H3-CO2H.

IT **24155-97-3P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
RN 24155-97-3 HCAPLUS
CN m-Toluoyl chloride, 6-[[m-(trifluoromethyl)benzyl]sulfonyl]- (8CI) (CA INDEX NAME)

Searched by Barb O'Bryen, STIC 308-4291



L36 ANSWER 19 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1963:454908 HCAPLUS
DOCUMENT NUMBER: 59:54908
ORIGINAL REFERENCE NO.: 59:10010e-h,10011a
TITLE: 11-(3-Dimethylaminopropylidene)-6,11-dihydrodibenz[b,e]-thiepin
INVENTOR(S): Protiva, Miroslav; Rajsner, Miroslav; Votava, Zdenek; Metysova, Jirina
SOURCE: 4 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 105590		19621115	CS	19610608

GI For diagram(s), see printed CA Issue.

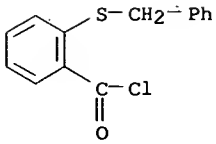
AB The title compd. (I) has thymoleptic, tranquilizing, antispasmodic, and antihistamine activity. S-Benzylthiosalicylic acid (II) (12.2 g.) in 70 ml. Et2O and 4 g. anhyd. C5H5N treated with 6 g. SOCl2 under cooling, the mixt. kept 2 hrs. at room temp., filtered, and the solid crystd. from C6H6-petr. ether gave the acid chloride (III), m. 118-19.degree.. II (40 g.), 110 g. P2O5 and 750 ml. anhyd. C6H6 refluxed 2 hrs., the mixt. kept overnight at room temp., decompd. by pouring into ice, the C6H6 layer sepd., dried (Na2SO4), and evapd. gave 6.7 g. acid anhydride (IV), m. 106-7.degree. (C6H6-petr. ether). Crude III (prepd. from 12.2 g. II) in 30 ml. PhNO2 treated under cooling and stirring with 12 g. AlCl3 in 30 ml. PhNO2, the mixt. kept 18 hrs. at room temp., poured into a mixt. of ice and dil. HCl, the org. layer sepd., washed (NaOH), dried (K2CO3), evapd. in vacuo, and distd. gave V, b0.1 162-5.degree., m. 85-6.degree. (Et2O-petr. ether). AlCl3 (50 g.) in 70 ml. PhNO2 treated with 41 g. IV in 130 ml. PhNO2 under cooling and stirring, the mixt. kept 20 hrs. at room temp., decompd. with ice and HCl, the org. layer sepd., washed, dried, and distd, gave V, b1 175-80.degree.. Me2N(CH2)3 MgCl [prepd. from 1.5 g. Mg, several drops of EtBr, and 9 ml. Me2N(CH2)3Cl in 30 ml. anhyd. Et2O] treated with 6.5 g. V in 25 ml. C6H6 under stirring, the mixt. refluxed 18 hrs., cooled, decompd. with 100 ml. 10% NH4Cl, dild. with 100 ml. CHCl3, the org. layer sepd., dried (K2CO3), and evapd. gave 9.0 g. 11-(3-dimethylaminopropyl)-6,11-dihydrodibenz[b,e]thiepin-11-ol (VI), m. 130-1.degree. (C6H6-petr. ether)., VI (8.0 g.) and 70 ml. 3N H2SO4 refluxed 5 min., the soln. filtered with C, made alk. with 20% NaOH, extd. with CHCl3, the ext. dried (K2CO3), evapd., and the residue distd. gave 4.3 g. I, b0.2 162-4.degree.; HCl salt m. 215-17.degree. (EtOH-Et2O).

IT **1531-81-3**, Benzoyl chloride, o-(benzylthio)-
(prepn. of)

RN 1531-81-3 HCAPLUS

CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

Searched by Barb O'Bryen, STIC 308-4291



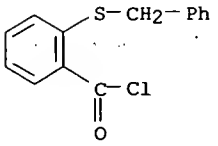
L36 ANSWER 20 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1963:27348 HCAPLUS
DOCUMENT NUMBER: 58:27348
ORIGINAL REFERENCE NO.: 58:4574c-h,4575a-h,4576a-d
TITLE: Synthetic medicinals. VIII. New-type tricyclic thiazepine and thiepin derivatives
AUTHOR(S): Gadient, F.; Jucker, E.; Lindenmann, A.; Taeschler, M.
CORPORATE SOURCE: Sandoz A.-G., Basel, Switz.
SOURCE: Helv. Chim. Acta (1962), 45, 1800-70
DOCUMENT TYPE: Journal
LANGUAGE: German
GI For diagram(s), see printed CA Issue.
AB cf. CA 56, 1532i. The syntheses and pharmacol. properties were described of new type tricyclic compds., derivs. of 5,11-dihydrobenzo[b]pyrido[2,3-e]-1,4-thiazepine (I) and of 6,11-dihydrodibenzo[b,e]thiepin (II). To 16.0 g. 3-hydroxymethylpyridine N-oxide in 75 ml. CHCl3 was added dropwise during 30 min. 43.0 g. SOCl2 under H2O cooling, the whole refluxed 2 hrs., and cooled in ice H2O to give 3-chloromethylpyridine N-oxide HCl salt (III.HCl), m. 98-100.degree. (CHCl3). III.HCl (9.0 g.) suspended in 60 ml. CHCl3, shaken with 4.2 g. NaHCO3 in 40 ml. H2O, the aq. phase sepd., extd. twice with 60 ml. CHCl3, the combined CHCl3 solns. dried, and concd. in vacuo until crystn. commenced gave III, m. 135-7.degree. (CHCl3). III (34.0 g.) added during 30 min. to 100 ml. POCl3 at 25-30.degree., the whole refluxed 2 hrs., the excess POCl3 completely removed in vacuo, the residue dissolved in 100 ml. CHCl3, the soln. washed with 100 g. ice H2O, dried, and fractionated gave 2-chloro-3-chloromethylpyridine (IV), b13 115.degree.. IV (8.1 g.) added rapidly dropwise to 6.25 g. 2-H2NC6H4SH and 2.0 g. NaOH in 40 ml. EtOH and 10 ml. H2O in an N atm., the whole refluxed 70 min., cooled, filtered, the filtrate concd. in vacuo, the residue dissolved in 100 ml. CHCl3, the soln. extd. with 2 50-ml. portions 5N HCl, the combined exts. neutralized with 5N NaOH, the product isolated with CHCl3, and distd. gave 2-chloro-3-[(2-aminophenyl)thiomethyl]pyridine (V), b0.02 150-60.degree. (air bath temp.). V (70.0 g.) and 6.0 g. PhNMe2 in 130 ml. xylene refluxed 4 hrs., the resulting ppt. filtered off, partitioned between 200 ml. CHCl3 and 100 ml. 10% aq. NaHCO3, the CHCl3 layer washed neutral with H2O, dried, and concd. deposited I, m. 123-5.degree. (C6H6). I (3.3 g.) and 900 mg. 50% NaH in oil suspension in 60 ml. xylene heated 2 hrs. at 160.degree., the whole treated dropwise during 1 hr. with 2.5 g. 2-(2-chloroethyl)-1-methyl-piperidine in 10 ml. xylene, kept 3 hrs. at 160.degree. cooled, treated with 3 g. NH4Cl in 30 ml. H2O, filtered through diatomaceous earth, the xylene layer in the filtrate sepd., washed with 50 ml. H2O, extd. with 100 ml. 15% aq. tartaric acid, the ext. washed with 20 ml. C6H6, made alk. with 5N NaOH, and the product isolated with C6H6 gave 11-[2-(1-methyl-2-piperidyl)ethyl] deriv. (VI) of I, oil, which was purified on Al2O3 with C6H6. Purified VI (3.4 g.) in 30 ml. MeOH treated with 3.8 g. (76% moist) 1,5-naphthalenedisulfonic acid in 5 ml. MeOH and 1 ml. H2O and kept at room temp. gave VI 1,5-naphthalenedisulfonate (VII) hydrate, m. 235-50.degree. (decompn.) (aq. MeOH). Similarly were prepd. 11-(3-dimethylaminopropyl) deriv. (VIII) of I 1,5-naphthalenedisulfonate, m. 175-85.degree. (decompn.) (aq. EtOH), and 11-(2-dimethylaminopropyl) deriv. (IX) of I 1,5-naphthalenedisulfonate, m. 170-80.degree. (decompn.) (aq. EtOH). 2-MeC6H4CO2Et (IXa), 107 g. SO2Cl2, and 760 mg. Bz2O2 heated

at 60.degree. (oil bath) while irradiating with ultraviolet light, when gas evolution stopped the unchanged IXa distd. in vacuo (at 13 mm.), and the residue fractionated gave 2-ClCH2C6H4CO2Et (X), b0.03 100-2.degree.. X (87.0 g.) added dropwise to 48.2 g. PhSH and 17.5 g. NaOH in 90 ml. H2O and 350 ml. EtOH, the whole refluxed 75 min., cooled, filtered, the filtrate concd. in vacuo, the residue dissolved in 300 ml. CHCl3, the soln. washed with 50 ml. ice cold N NaOH and with H2O until neutral, dried, and fractionated gave 2-(4-RC6H4SCH2)C6H4CO2R' (XI) (R = H, R' = Et), b0.2 140-2.degree.. The following XI (R' = Et) were similarly prepd. (R and b.p./mm. given): Cl, 176-8.degree./0.1: Me, 145-50.degree./0.02; MeO, 175-80.degree./ 0.05; MeS, 160.degree./0.01; F3C (prepd. from 4-F3CC6H4SH, b13 60-1.degree., which was prepd. from 4-F3CC6H4SO2Cl, b0.03 56-60.degree., m. 31-3.degree., which was obtained from 4-F3CC6H4NH2), 118-20.degree./ 0.02.. XI (R = H, R' = Et) (78.0 g.) boiled 1 hr. with 13.0 g. NaOH in 78 ml. H2O and 53 ml. EtOH, the soln. concd. in vacuo, dild. with 200 ml. H2O, washed with 50 ml. CHCl3, acidified with 5N HCl, extd. with 1200 ml. CHCl3, the ext. washed with H2O, dried, concd. somewhat, and dild. with petr. ether gave XI (R = R' = H), m. 111-13.degree. (CHCl3-petr. ether). The following XI (R' = H) were prepd. similarly (R, m.p., and recrystn. solvent given): Cl, 134-5.degree., CHCl3-pentane; Me, 130-1.degree., EtOH-pentane: MeO, 124-6.degree., EtOH-pentane; MeS, 135-7.degree., EtOH-pentane; F3C, 125-8.degree., EtOH-pentane.. XI (R = R' = H) (50.0 g.) heated 20 min. at 60.degree. with 200 g. SOCl2 and the product fractionated gave 2-(4-RC6H4SCH2)C6H4COCl (XII) (R = H), b0.1 165-7.degree.. Similarly was prepd. XII (R = Cl), b0.1 178-80.degree.. Method A. XII (R = H) (10.0 g.) in 70 ml. CS2 added dropwise during 30 min. to 10.0 g. AlCl3 suspended in 30 ml. boiling CS2, after 15 hrs. the CS2 removed, the residue treated with 50 g. ice and 15 ml. concd. HCl under cooling, extd. with 100 ml. Et2O, the ext. washed with 30 ml. 2N NaOH and with H2O until neutral, dried, concd., the crude product boiled in EtOH with C, and the EtOH soln. concd. deposited 11-oxo deriv. (XIII) of II, m. 84-6.degree. (EtOH); better yields were obtained by method B. Method B. To 207 ml. 85% H3PO4 was added 300 g. P2O5 at 80-100.degree. with stirring, the polyphosphoric acid mixt. kept at 100.degree., treated during 10 min. with 105.0 g. XI (R = Me, R' = H), stirred 75 min. at 100.degree., poured while hot onto 1 kg. ice with stirring, treated with 600 ml. C6H6, filtered through diatomaceous earth, the C6H6 layer in the filtrate sepd., the aq. layer extd. twice with 200 ml. C6H6, the combined C6H6 solns. extd. washed with 3 100-ml. portions 2N NaOH and with H2O until neutral, dried, concd., the residue dissolved in boiling EtOH, the soln. treated with C, and cooled to give 2-Me deriv. of XIII, m. 121-2.degree. (EtOH).. Method C. XI (R = MeO, R' = H) (100.0 g.) added to 300 g. P2O5 and 200 ml. 85% H3PO4 in 2 l. PhMe at the b.p. with stirring, the mixt. heated 17 hrs., the PhMe soln. decanted while hot, the residue extd. with 4 1-l. portions boiling PhMe, the combined PhMe solns. washed with 11.2N NaOH and with H2O until neutral, dried, concd. in vacuo, the residue dissolved in boiling EtOH, the soln. treated with C, and cooled gave 2-MeO deriv. of XIII, m. 94-6.degree.. The following 2-substituted derivs. of XIII were also prepd. (2-substituent, method, and m.p. given): Cl (XIV), B, 134-6.degree. (EtOH); MeS, C, 92-4.degree. (EtOH); F3C, B, 116-19.degree.. Iodine-activated Mg (1.1 g.) covered with a little tetrahydrofuran, treated with 0.1 ml. (BrCH2)2, when the reaction commenced the mixt. treated dropwise with 5.4 g. Me2N(CH2)3Cl in 10 ml. tetrahydrofuran in such a manner that the solvent boiled, refluxed 2 hrs., treated during 10 min. with 5.2 g. XIV in 15 ml. tetrahydrofuran, boiled and stirred 10 min., cooled, poured into 100 ml. H2O contg. 15 g. NH4Cl, treated with 100 ml. Et2O, filtered through diatomaceous earth, the Et2O layer in the filtrate sepd., the aq. layer extd. with 3 50-ml. portions Et2O, the combined Et2O solns. washed with H2O, dried, evapd., the oily residue dissolved in 10 ml. Me2CO, and the soln. kept gave 2-chloro-11-(3-dimethylaminopropyl)-11-hydroxy-6,11-dihydrodibenzo[b,e]thiepin (XV [R = Cl, R' = Me2N(CH2)3]) (XVa), m. 154-5.degree. (EtOH-pentane).. XVa (5.0 g.) in 50 ml. AcOH boiled 1 hr.

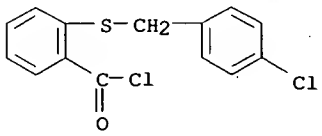
with 20 ml. concd. HCl, evapd. in vacuo (15 mm.), the residue made alk. with 2N NaOH, extd. with 3 50-ml. portions CHCl3, the combined exts. washed with H2O, dried, and evapd. gave 2-chloro-11-(3-dimethylamino-propylidene)-6,11-dihydrodibenzo[b,e]thiepin [XVI (R = Cl, R' = Me2NCH2-CH2CH)], oil; oxalate m. 215-16.degree. (EtOH). The following addnl. XV were prepd. (R, R', and m.p. given): H, 1-methyl-4-piperidyl, 184-7.degree.; H, 2-(1-methyl-2-piperidyl)-ethyl, 175-84.degree.; H, Me2N(CH2)3, 130-2.degree.; H, Et2N(CH2)3, 105-7.degree.; H, 3-(1-piperidyl)propyl, 190-2.degree.; H, 3-(1-morpholinyl)-propyl, 175-7.degree.; H, 3-(1-morpholinyl)-2-methylpropyl, 163-5.degree.; H. 1-methyl-3-piperidylmethyl, 170-5.degree.; H, 3-(1-piperidyl)-2-methylpropyl, 187-9.degree.; H, 1-methyl-3-pyrrolidylmethyl, -- (b0.15 200.degree.); H, 2-(1-methyl-2-pyrrolidyl)ethyl, 192-200.degree. and 116-20.degree. (2-isomers were isolated, in all other cases only 1 isomer was isolated); Cl, 1-methyl-4-piperidyl, 182-4.degree.; Cl, 3-(1-piperidyl)propyl, 195-7.degree.; Cl, 2-(1-methyl-2-piperidyl)ethyl, oil; Me, 1-methyl-4-piperidyl, 181-3.degree.; Me, Me2N(CH2)3, 139-42.degree.; MeS, 1-methyl-4-piperidyl, 178-80.degree.; MeS, Me2N(CH2)3, 137-8.degree.; MeO, 2-(1-methyl-2-piperidyl)ethyl, 141-2.degree.; MeO, Me2N(CH2)3, 123-5.degree.; MeO, 1-methyl-4-piperidyl, 182-5.degree.. The XV were not tested since previous experiences had shown them to have only slight activity. The following XVI were prepd. and tested [R, R', m.p., % histamine inhibition (thenalidine = 100%) (effective concn.: 5 .times. 10-8), % acetylcholine inhibition (atropine = 100%) (effective concn.: 1 .times. 10-9) given]: H, 1-methyl-4-piperidylidene (XVII), -- [HBr salt m. 265-70.degree. (decompn.)], 200, 33; H, 2-(1-methyl-2-piperidyl)ethylidene, -- [HBr salt m. 210-17.degree. (decompn.)], --, --; H, Me2NCH2CH2CH, -- (oxalate m. 167-9.degree.), 25, 10; H, Et2NCH2CH2CH, -- (oxalate m. 174-6.degree.), 33, 5; H, 3-(1-piperidyl)propylidene, -- (fumarate m. 193-7.degree.), 33,5; H, 3-(1-morpholinyl)propylidene, -- (fumarate m. 165-8.degree.), 50, 1.7; H, 3-(1-morpholinyl)-2-methyl-propylidene, -- (fumarate m. 182-5.degree.), 3.3, 0.5; H, 1-methyl-3-piperidylmethylene, -- (fumarate m. 240-2.degree.), 17, 10; H, 3-(1-piperidyl)-2-methylpropylidene, -- (oxalate m. 187-9.degree.), 10, 0.17; H, 1-methyl-3-pyrrolidylmethylene, -- (fumarate m. 213-15.degree.), 200, 17; 2-(1-methyl-2-pyrrolidyl)ethylidene, -- (oxalate m. 150-3.degree.), 400, 33; Cl, 1-methyl-4-piperidylidene, 161-4.degree., 200, 20; Cl, Me2NCH2CH2CH, -- (oxalate m. 215-16.degree.), 100, 2; Cl, 3-(1-piperidyl)propylidene, -- (fumarate m. 240-5.degree.), 7, 1.7; Cl, 2-(1-methyl-2-piperidyl)ethylidene, -- [HBr salt m. 245-60.degree. (decompn.)], 50, 6.5; Me, 1-methyl-4-piperidylidene, -- (HBr salt m. 294-7.degree.), 67, 3.3; Me, Me2NCH2CH2CH, -- (oxalate m. 189-92.degree.), 67, 3.3; MeS, 1-methyl-4-piperidylidene, 154-5.degree., 100, 4; MeS, Me2NCH2CH2CH, -- (oxalate m. 180-5.degree.), 100, 1.3; MeO, 2-(1-methyl-2-piperidyl)ethylidene, -- (HCl salt m. 204-11.degree.), 50, 5; MeO, Me2NCH2CH2CH, -- (oxalate m. 187-9.degree.), 100, 1.3; MeO, 1-methyl-4 piperidylidene, 120-1.degree., 100, 10. The I series showed weak activity as follows [compd., % histamine inhibition (thenalidene 100%), and % acetylcholine inhibition (atropine = 100%)given]: VII, 2, 6; VIII, 1, 7; IX, 18, 2. The pharmacol. properties of XVII.HBr were more fully investigated. The antihistamine action of XVII.HBr was appreciably more pronounced in whole animal than in the in vitro studies. Thus 10-100 .gamma. XVII.HBr/kg. intravenously was able to arrest the blood pressure lowering effect of histamine in anesthetized cats. Subcutaneous doses of 0.15-0.3 mg. XVII.HBr/kg. prevented up to 50% the lethal and bronchoconstrictor action of histamine in guinea pigs. In these investigations in whole animals XVII.HBr was 20-30 times more effective than thenalidine. XVII.HBr also showed strong serotonin inhibiting action in the isolated rat uterus. It lacked any appreciable sedative effects.

IT **1531-81-3**, Benzoyl chloride, o-(benzylthio)- **92153-07-6**, Benzoyl chloride, o-[(p-chlorobenzyl)thio]-
(prepn. of)

RN 1531-81-3 HCAPLUS
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



RN 92153-07-6 HCAPLUS
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)



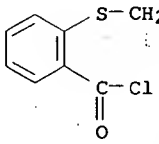
L36 ANSWER 21 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1963:415510 HCAPLUS
DOCUMENT NUMBER: 59:15510
ORIGINAL REFERENCE NO.: 59:2772g-h,2773a-f
TITLE: Synthetic ataractics. VII. 11-(3-Dimethylaminopropylidene)6,11-dihydrodibenzo[b,e]thiepin
AUTHOR(S): Rajsner, M.; Protiva, M.
CORPORATE SOURCE: Pharm. Res. Inst., Prague
SOURCE: Cesk. Farm. 11 (1962), 404-9
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. CA 57, 9817e; 58, 7853g. S-Benzylthiosalicylic acid (I) (40 g.), m. 189.degree., 110 g. P2O5, and 750 ml. anhyd. C6H6 refluxed 2 hrs., the mixt. kept overnight at room temp., decompd. by pouring onto ice, the org. layer sepd., the aq. layer extd. with C6H6, and the org. solns. combined, dried (Na2SO4), and evapd. to dryness gave 29 g. S-benzylthiosalicylic acid anhydride (II), m. 107-7.5.degree. (C6H6-petr. ether). Hydrolysis of II with boiling NaOH in aq. EtOH gave I. I (10 g.) and 25 ml. SOCl2 refluxed till the evolution of gaseous products ceased, the mixt. evapd. in vacuo to dryness, and the residue mixed with EtOH gave 5.8 g. bis(thiosalicylic acid) dichloride, m. 159-61.degree. (CHCl3-petr. ether). I (12.2 g.) in 70 ml. Et2O treated with 4 ml. anhyd. C5H5N and then treated under cooling and shaking with 6 g. SOCl2, the mixt. kept 2 hrs. at room temp. and dild. with petr. ether, the solid filtered off, and the filtrate extd. with 200 ml. C6H6, the ext. filtered, and the soln. evapd. in vacuo to dryness gave 6.5 g. S-benzylthiosalicylic acid chloride (III), m. 117-19.degree. (C6H6-petr. ether), ν (Nujol) 710, 750-80, 1260-75, 1465, 1495-1570-90, 1680 cm.-1 EtONa (prepd. from 92 g. Na and 1400 ml. anhyd. EtOH) treated with 440.3 g. PhSH and 536.5 g. phthalide, the mixt. refluxed 4.5 hrs., the greater part of EtOH distd. in vacuo, the residue dissolved in 3 l. H2O, the soln. filtered, the filtrate cooled, and acidified with HCl gave 920 g. o-(phenylthiomethyl)- benzoic acid (IV), m. 113-16.degree. (80% EtOH). IV (24.4 g.) and 50 ml. SOCl2 kept 20 min. at room temp., the mixt. heated to 60.degree. till evolution of gaseous products ceased and evapd. in vacuo, and the residue distd. gave 17 g. acid chloride of IV, b0.5 142-50.degree.. AlCl3 (50 g.) in 70 ml. PhNO2

cooled with ice, treated dropwise with stirring with 41 g. II in 130 ml. PhNO2, the mixt. kept 20 hrs. at room temp., poured onto ice and dil. HCl, the org. layer sepd., washed (dil. HCl, dil. NaOH), dried (K2CO3), evapd. in vacuo to dryness, and the residue distd. gave 5.3 g. 6,11-dihydrodibenzo[b,e]thiepin-11-one (V), b1 175-80.degree., m. 80-7.degree. (Et2O-petr. ether), v (CCl4, Nujol) 703, 733, 759, 777, 800, 930, 1045, 1072, 1118, 1152, 1249, 1291-1300, 1428, 1452, 1463, 1595, 1652 cm.-1 III (6.5 g.) in 30 ml. PhNO2 treated under external cooling dropwise with 12 g. AlCl3 in 30 ml. PhNO2, the mixt. kept 18 hrs. at room temp., and worked up gave 1.4 g. V, b0.1-162-5.degree., m. 86-7.degree.. IV (160 g.) cyclized 1 hr. with polyphosphoric acid (prepd. from 510 g. P2O5 and 340 ml. 90% H3PO4) at 90.degree., the mixt. poured onto 2 kg. ice and H2O and extd. with C6H6, and the org. layer washed (H2O, 5% NaOH), dried (K2CO3), and evapd. gave 113.5 g. V, m. 86-7.degree. (EtOH). V (2.3 g.) in 30 ml. anhyd. MeOH reduced with 0.6 g. NaBH4, the mixt. refluxed 10 min. and evapd., the residue decompd. with 20 ml. H2O, extd. with CHCl3, and the ext. dried (MgSO4) and evapd. gave 2.1 g. 6,11-dihydrodibenzo[b,e]thiepin-11-ol, m. 107-8.degree. (C6H6-petr. ether). V (2.3 g.) in 15 ml. AcOH treated with 1 ml. 30% H2O2, the mixt. kept 48 hrs. at room temp., and dild. with 70 ml. H2O gave 2.0 g. 6,11-dihydrodibenzo [b,e] thiepin-11-one 5-oxide, m. 97-100.degree. (EtOH). V (2.3 g.) in 15 ml. AcOH- treated with 4.6 ml. 30% H2O2 and the mixt. refluxed 3 hrs. and cooled gave 2.15 g. 6,11-dihydrodibenzo [b,e]thiepin-11-one 5,5dioxide, m. 127-8.degree. (EtOH). Me2N(CH2)3MgCl [from 38.6 g. Mg, 5 ml. EtBr, and 193 g. Me2N(CH2)3Cl in 600 ml. anhyd. Et2O] refluxed and treated dropwise with 185 g. V in 750 ml. C6H6, the mixt. stirred and refluxed 18 hrs., cooled, and decompd. with 1500 ml. 10% NH4Cl, the org. layer sepd., dried (K2CO3), and partially evapd., and the residue treated with 500 ml. petr. ether gave 154 g. 11-(3-dimethylaminopropyl) 6,11-dihydrodibenzo[b,e]thiepin-11-ol (VI), m. 130-1.degree. (C6H6-petr. ether), .lambda. 261 m.mu. (log .epsilon. 4.0) in MeOH, v (CHCl3) 770-90, 1110-70, 1430, 1460, 1590, 2780-2825 cm.-1 VI (130 g.) and 1000 ml. 3N H2SO4 refluxed 20 min., the mixt. cooled, made alk. with 25% NaOH, and extd. with Et2O, the ext. dried (K2CO3) and evapd., and the residue (120.5 g.) dissolved in 100 ml. anhyd. EtOH and acidified with anhyd. HCl in Et2O gave 123 g. HCl salt of VII, m. 218-21.degree. (EtOH-Et2O), .lambda. 232, 260, 309 m.mu. (log .epsilon. 4.41, 3.97, 3.53) in MeOH, v (CHCl3) 760-90, 1430, 1460, 1590, 2350, 3400 cm.-1; the base b0.2 162-4.degree.. The HCl salt of VII (prothiadene) has mild tranquilizing activity and is being clinically tested as an antidepressive drug.

IT **1531-81-3**, Benzoyl chloride, o-(benzylthio)-
(prepn. of)

RN 1531-81-3 HCAPLUS

CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 22 OF 32 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1962:24876 HCAPLUS

DOCUMENT NUMBER: 56:24876

ORIGINAL REFERENCE NO.: 56:4664g-i,4665a-i,4666a-h

TITLE: Dialkylaminoalkyl N- or S-derivatives of 2-mercapto-2,2'-dithio, 2-(alkylthio)-, 2-(aralkylthio)-, and 2-(arylthio)benzamides

Searched by Barb O'Bryen, STIC 308-4291

AUTHOR(S): Gialdi, F.; Ponci, R.; Baruffini, A.
CORPORATE SOURCE: Univ. Pavia, Italy
SOURCE: Farmaco (Pavia) Ed. Sci. (1961), 16, 411-37
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB The high antifungal activity in vitro found previously (CA 55, 21040b) in aromatic disulfides and o-earbamoyl substituted sulfides prompted the synthesis of [o-R2N(CH2)nHNCOC6H4]2S (I), o-R'SC6H4CONH(CH2)nNR2 (II), and o-R2N(CH2)nSC6H4CONHR' (III). A soln. of 0.015 mole 2,2'-dicarboxydiphenyl disulfide (IV) in 100 ml. C6H6 was added slowly and under stirring to 0.03 mole of the appropriate diamine in 50 ml. C6H6. The mixt. was kept overnight at room temp., then cooled, extd. with dild. HCl (50 ml.), the acid layer decolored with charcoal, filtered and neutralized with dild. NaOH, to give a product which solidified on standing in a refrigerator. After filtration and crystn. the following I were obtained (n, R, m.p., and crystn. solvent given): 2, Et (V), 135.degree., dil. al.; 3, Et (VI), 114-15.degree., benzene-petr. ether. By adding a soln. of 0.06 mole H2N(CH2)3NMe2 in 15 ml. dioxane to a soln. of 0.015 mole IV in 70 ml. dioxane, heating the mixt. 15 min. at 70.degree. with stirring, then cooling, adding 150 ml. petr. ether, cooling, and filtering, a cryst. product was collected, which was dissolved in 50 ml. dild. HCl, the soln. filtered through charcoal, cooled, basified with satd. Na2CO3 soln., and the ppt. collected and crystd. from Me2CO3 to give I (n = 3, R = Me) (VII), m. 42.degree.. VI.2MeI m. 1869.degree. (EtOH). The following II were prepd. by reaction of an appropriate diamine with o-R'SC6H4COCl (VIII), according the procedure described for VIII (n,R,R',m.p., and crystn. solvent given): 2, Et, Et, 115-17.degree. (as HCl salt), MeOH-ether; 2, Et, Bu, 95.degree. (as HCl salt), Me2COether; 2, Et, isoamyl, 40.degree., dil. Me2CO; 2, Et, p-O2NC6H4, 89-90.degree., dil. EtOH; 2, Et, PhCH2, 132-4.degree., MeOH-ether; 3, Me, PhCH2 (IX), 81.degree., dil. MeOH; 2, Et, p-O2NC6H4CH2, 134-6.degree. (as HCl salt), Me2CO-MeOH-ether; 3, Et, p-O2NC6H4CH2, 69-70.degree., dil. EtOH; 2, Et, p-ClC6H4CH2, 178-9.degree. (as HCl salt), EtOH-ether; 3, Me, p-ClC5H4CH2 (X), 89.degree., ether-petr. ether; 2, Et, p-MeOC6H4CH2, 46-8.degree., ether-petr. ether. IX.MeI (XI), m. 119.degree. (EtOH-ether); X.MeI (XII) m. 136-5.degree. (EtOH-ether). VIII were prepd. by reaction of SOCl2 with the corresponding carboyxlic acid in ether or without solvent. The prepn. of the unknown 2-(isoamylthio)benzoic acid (XIII) was reported. Thus, to 0.1 mole thiosalicylic acid (XIV) in 40 ml. H2O and 0.2 mol. 20% KOH, heated at 70.degree., 0.1 mole isoamyl bromide dissolved in 100 ml. EtOH was added and the mixt. refluxed 2 hrs. with stirring. The resulting soln. was coned, to half-vol., dild. with 50 ml. H2O, filtered through charcoal, and acidified with dil. HCl to give an oil which solidified on cooling. The solid was collected and crystd, from EtOH and from ether-ligroine to give XIII, m. 86-7.degree.; the corresponding acid chloride (XV) (VIII, R' = isoamyl), obtained in ether soln. from XIII and SOCl2, was characterized through the anilide, m. 78.degree. (80% EtOH). An improved synthesis of 2-(4-nitrophenylthio)benzoic acid (XVI) was described. To 0.2 mole XIV and 0.22 mole anhyd. K2CO3 in 200 ml. H2O at 80% 2 g. KI was added, and then with stirring, a 0.2 mole of 4-chloronitrobenzene in 360 ml. EtOH was added. The mixt. was refluxed 5 hrs., then coned, to half-vol., the soln. refluxed 5 hrs., coned, to 2/3 vol. and refluxed 5 hrs. After cooling, the reaction mass was poured into lb0 g. ice and acidified with dild. HCl, the ppt. collected, washed with H2O and crystd, from EtOH to give 88% XVI, m. 229-30.degree.; the corresponding acid chloride (XVII) (VIII, R' = p-O2NC6H4) m. 129-30.degree. (C6H6-ligroine). XVII was further characterized through 2-(4-nitrophenylthio)benzamide (XVIII), m. 172-4.degree. (EtOH). XVI, when refluxed 15 min. with POCl3, gave a mixt. of XVII and 2-nitrothioxanthone (XIX), m. 220-2.degree. (AcOH); when the heating was prolonged for 1 hr., only XIX was isolated. To study the biol. variations in I and II in which the secondary amide was substituted

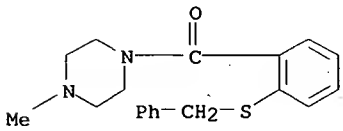
by a tertiary amide, a no. of N-methylpiperazides of I and II was prepd. Thus, 3.5 g. IV in 45 ml. dioxane, treated with 2 g. N-methylpiperazine (XX) at 20-25% the mixt. kept 2 hrs. at room temp., 60 ml. Et2O added, and the crystals crystd. from EtOH-ether, gave the N-methylpiperazide of 2,2'-dicarboxyldiphenyl disulfide-2HCl (XXI), m. 225-8.degree. (decompn.); dimethiodide (XXII) m. 246% Similarly, 0.02 mole of appropriate VIII in 50 ml. dioxane, added to 0.045 mole XX in 10 ml. dioxane, the mixt. heated 10 min. at 50.degree., 3 vols. H2O added, the oil sepd. and extd. with ether, washed with dild. NaHCO3, then with H2O, the ext. dried over Na2SO4, the solvent evapd. gave the following XXIII (R', m.p. and crystn. solvent of HCl salt, and m.p. and crystn, solvent of methiodide given): isoamyl, 210-14.degree., EtOH-Et2O, 109.degree., EtOH-Et2O; p-O2NC6H4, 236.degree., EtOH-Et2O, -, -; PhCH2, 198.degree., MeOH-Et2O, 183-4.degree., EtOH-Et2O; p-ClC6H4- CH2, 140-1.degree., EtOH-Et2O, -, -. For comparison with the parent I and II, a no. of .beta.-diethylaminoethyl esters was prepd. Thus, 1 mole IV in dioxane added to a dioxane soln. of .beta.-diethylaminoethanol (XXIIa), the mixt. heated 20-30 min. at 50-60.degree., then kept some hrs. at room temp., 5-6 vols. H2O added, sepd. an oil which extd. with ether, washed with H2O, dried and satd. with dry HCl gave (o-Et2NCH2CH2O2CC6H4S)2.2HCl (XXIII), m. 186-8.degree. (MeOH-Et2O). The following o-R'SC6H4COOCH2CH2NEt2.HCl (XXIV) were obtained from the appropriate VIII and XXIIa (R', m.p., and crystn. solvent given): Et, 127-8.degree., Me2CO-Et2O; Bu, 117.degree., Me2CO-Et2O; p-O2NC6H4, 55-6.degree. (as base), ligroine; PhCH2, 144-5.degree., MeOH-Et2O; p-O2NC6H4CH2, 173.degree.-5.degree., MeOH-Et2O; p-MeOC6H4CH2, 67-9.degree. (as base), ligroine. To 0.02 mole 2-mercaptobenzamide (XXV), and 0.92 mole ClCH2CH2NMe2.HCl (XXVI) in 30 ml. EtOH, was added dropwise with stirring under N a soln. of EtONa (from 0.92 g. Na in 15 ml. EtOH), and the temp. of the soln. was slowly raised to reflux. After refluxing 45 min., the mixt. was cooled, NaCl filtered off, the filtrate evapd., the residue dissolved in 30 ml. hot H2O, basified with NH4OH satd. on cooling with AcONa, and the ppt. collected, dried and crystd. several times from C6H6ligroine to give III (R = Me, R' = H, n = 2) (XXVII), m. 105.degree.; methiodide m. 190.degree. (EtOH-Et2O); method A. 2-Mercaptobenzanilide (XXVIII) (0.02 mole), 0.02 mole XXVI, and 0.01 mole K2CO3 refluxed 1 hr. in 30 ml. EtOH, the ale. soln. coned., dild. with 6 vols. H2O, kept overnight in a refrigerator, the ppt. collected, dissolved in dild. HCl, the soln. filtd. through charcoal, basified with NaOH, the ppt. filtered and crystd. from dild. alc., then from ligroine, gave III (R = Me, R' = Ph, n = 2) (XXIX), m. 94.degree.; methiodide m. 213.degree. (EtOH); methobromide m. 172.degree. (EtOH); method B. A soln. of 0.01 mole 2-(.gamma.-chloropropylthio)benzanilide (XXX) and 0.05 mole HNEt2 in 15 ml. EtOH was refluxed 12 hrs., then cooled, dild. with 10 vols. ice H2O, the ppt. filtered off, dissolved in dild. HCl, the soln. filtered through charcoal, basified on cooling with NaOH, the ppt. filtered off washed with H2O, dried and crystd. from ligroine gave 68% III (R = Et, R' = Ph, n = 3) (XXX1), m. 70.5.degree.; method C. The starting XXX was prepd. as follows. Thiosalicylic acid (15.4 g.) suspended in 50 ml. EtOH, treated with 13.8 g. K2CO3 in 25 ml. H2O and with 15.7 g. 1-bromo-3-chloropropane was heated 10 min. at 50.degree., the resulting soln. cooled, poured into 3 vols. H2O, acidified, and the ppt. collected and crystd. twice from dild. alc. gave 74% 2-(.gamma.-chloropropylthio)benzoic acid (XXXII), m. 128-9.degree., which refluxed 1 hr. in excess SOCl2 was transformed to the corresponding crude acid chloride (XXXIII). The latter (0.01 mole) dissolved in 12 ml. dioxane, the mixt. kept 2 hrs. at room temp., dild. with 1% HCl, cooled, the ppt. filtered off, washed with H2O and crystd. from dild. alc., then from C6H6-petr. ether, gave XXX, m. 99.5-101.degree.. The following III were also synthesized (method, NR2, R', n, m.p. of the base and crystn. solvent, m.p. of the methiodide and crystn. solvent given): B, C (from XXX), NMe2, Ph, 2, 81-4.degree., Et2O-petr. ether, -, -; A, piperidino, H, 2, 115.degree., Me2COpetr. ether, 184.degree., EtOH-Et2O; C (from XXX), piperidino, Ph, 3, 106.degree., dil. EtOH, -; -; A [from

N-(2-mercaptobenzoyl)-4-methoxyaniline (XXXIV)], NMe2, p-MeOC6H4, 3, 85.degree.. dil. EtOH, -, -; B, piperidino, p-MeOC6H4, 2, 109.degree., dil. EtOH, -, -; B [from N-(2-mercaptobenzoyl)4-chloroaniline (XXXV)], NMe2, p-ClC6H4, 3, 106, dil. EtOH, -, -; B, piperidino, p-ClC6H4, 2, 113.degree., dil. EtOH, -, -. XXXIV was prepd. by treating 5 g. of the bis(4 methoxyanilide) of IV (XXXVI) in 60 ml. EtOH with 6 g. Zn and 15 ml. concd. HCl, refluxing the mixt. to soln. of XXXVI, then filtered through Zn dust. The soln. was cooled, dil. with 3 vols. ice H2O, and the ppt. collected and crystd. from dil. AcOH to give the XXXIV, m. 136-7.degree.. Similarly, XXXV, m. 124-5.degree. (AcOH), was prepd. from the bis(4chloroanilide) of IV. All the products were tested in vitro on representative fungal strain and were found slightly active or inactive, thus giving evidence of the neg. influence regarding antifungal activity of the dialkylaminoalkyl group in the synthesized mols.

IT **98883-91-1**, Piperazine, 1-[o-(benzylthio)benzoyl]-4-methyl-, hydrochloride **98963-55-4**, Piperazine, 1-[o-[(p-chlorobenzyl)thio]benzoyl]-4-methyl-, hydrochloride
(prepn. of)

RN 98883-91-1 HCAPLUS

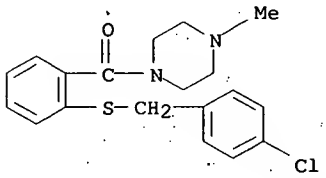
CN Piperazine, 1-[o-(benzylthio)benzoyl]-4-methyl-, hydrochloride (7CI) (CA INDEX NAME)



● HCl

RN 98963-55-4 HCAPLUS

CN Piperazine, 1-[o-[(p-chlorobenzyl)thio]benzoyl]-4-methyl-, hydrochloride (7CI) (CA INDEX NAME)



● HCl

L36 ANSWER 23 OF 82 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1961:111931 HCAPLUS

DOCUMENT NUMBER: 55:111931

ORIGINAL REFERENCE NO.: 55:21040b-i,21041a-f

TITLE: 2-Benzylthiobenzamides with antifungal activity

AUTHOR(S): Gialdi, F.; Ponci, R.; Baruffini, A.

CORPORATE SOURCE: Univ. Pavia, Italy

SOURCE: Farmaco (Pavia), Ed. sci. (1960), 15, 856-82

Searched by Barb O'Bryen, STIC 308-4291

DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

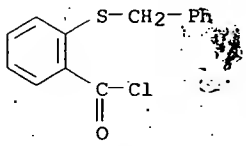
AB 2-(Benzylthio)benzoic acid (24.4 g.) in 240 cc. C6H6 treated with 24 g. SOCl2, refluxed 2 hrs., treated with 240 cc. ligroine, cooled, and filtered yielded 85-90% 2-(benzylthio)benzoyl chloride (I), m. 121-2.degree.. I(1 g.) boiled 1 hr. with 7 g. anhyd. MeOH gave Me 2-(benzylthio)benzoate (II), m. 67.degree.. I (2.6 g.) in 40 cc. dioxane basified with NH3 gas, dild. with 120 cc. ice H2O, neutralized with AcOH, the ppt. filtered off, washed with H2O, and crystd. from EtOH yielded 87% 2-(benzylthio)benzamide (III), m. 154-5.degree.. III was obtained also from thiosalicylamide and benzyl chloride. Aniline (0.04 mole) in 30 cc. dioxane, treated dropwise with 0.02 mole I in 70 cc. dioxane, heated 30 min. at 50-60.degree., cooled, dild. with 150 cc. H2O, acidified with HCl, the soln. filtered and the ppt. crystd. from EtOH yielded 2-(benzylthio)benzanilide (IV), m. 122.degree.. Similarly N-butyl-2-(benzylthio)benzamide (V), m. 91-2.degree., was prepd. N-(Benzyl)thiosalicylamide (VI), m. 110.degree., was synthesized by treating 5 g. bis(benzylamide) of 2,2'-dicarboxydiphenyl disulfide (VII) in 50 cc. EtOH with 5 cc. concd. HCl and 6 g. Zn. VII was prepd. by oxidn. with 0.5% H2O2 of VI in NaOH. VI (0.5 g.), treated with a stoichiometric amt. of 0.5N NaOH and 0.25 g. PhCH2Cl in 10 cc. EtOH, the mixt. heated 15 min. at 50.degree. and cooled, yielded 0.3 g. 2-benzylthio-N-benzylbenzamide (VIII), m. 102-3.degree.. The hydrolysis of VIII with 10% NaOH gave 2-(benzylthio)benzoic acid, m. 189.degree.. VI and VII in EtOH refluxed 4 hrs. with Raney Ni gave N-benzylbenzamide. VIII was obtained also from VII by condensing with PhCH2Cl with K2CO3 and refluxing 15 hrs. with PhCH2NH2. By the same method as for IV, the N,N-diethyl-2-(benzylthio)benzamide (IX), m. 81.degree. was prepd. N-[2-(Benzylthio)benzoyl]morpholine (X), m. 114.degree., and N-[2-(benzylthio)benzoyl] piperidine, m. 117-18.degree., were synthesized by the same method as for V. II, refluxed 2 hrs. with 7 cc. 95% hydrazine gave 2-(benzylthio)benzohydrazide, m. 164.degree.. Me thiosalicylate (16.8 g.) in 150 cc. EtOH, treated with 16.1 g. p-chlorobenzyl chloride (XI) with 6.9 g. K2CO3, the mixt. refluxed 1 hr., cooled, the soln. poured into 2 vols. ice H2O, and the ppt. filtered off and crystd. from EtOH yielded Me p-chlorobenzylthiobenzoate (XII), m. 102-3.degree.. 2-(4-Chlorobenzylthio)benzoic acid (XIII), m. 216-17.degree., was obtained by condensing thiosalicylic acid (XIV) and XI, in the presence of K2CO3 or boiling XII with concd. HCl. XIV (3.08 g.) in 30 cc. EtOH treated with 6.44 g. XI with 2.7 g. K2CO3, the mixt. refluxed 1 hr., the suspension dild. twice with ice H2O, filtered and the ppt. crystd. from acetone yielded 4-chlorobenzyl 2-(4 chlorobenzylthio)benzoate (XV), m. 166-7.degree.. XV boiled 5 hrs. with 1:1 EtOH- 10% NaOH gave XIII. 2-(4-Chlorobenzylthio)benzoyl chloride (XVI), m. 108-10.degree., was prepd. by the method as for I and the Me ester (XVII), m. 84.degree., was obtained from XVI as for II. 2-(4-Chlorobenzylthiobenzamide (XVIII), m. 147-8.degree., 2-(4-chlorobenzylthio)benzanilide (XIX), m. 127-8.degree., and 2-(4-chlorobenzylthio)-N-butylbenzamide, m. 98-100.degree., were prepd. As for IV, 2-(4-chlorobenzylthio)-N-benzylbenzamide, m. 130.degree., and 2-(4-chlorobenzylthio)-N,N-diethylbenzamide, m. 76-7.degree., were obtained. N-[2-(4-Chlorobenzylthio)benzoyl] morpholine (XX), m. 68-9.degree., and N-[2-(4-chlorobenzylthio)benzoyl]piperidine (XXI), m. 72-4.degree., were synthesized. The hydrazide (XXII) of 2-(4-chlorobenzylthio)benzoic acid, m. 166.degree., was obtained by boiling 5 hrs. under pressure 5 g. XVII and 1.5 cc. 95% hydrazine. 2-(4-Methoxybenzylthio)benzoic acid (XXIII), m. 218-19.degree., was obtained. p-Methoxybenzyl alcohol (40 g.), cooled on ice, treated dropwise with stirring with 50 g. SOCl2 during 20 min., the mixt. heated 1 hr. at 40.degree., cooled, treated with 2 g. CaCO3 and 60 cc. anhyd. Et2O, stirred several hrs., and finally kept 12 hrs. at room temp. yielded, after filtration and evapn. of Et2O and SOCl2, an oil, b5.0 98-102.degree., identified as p-methoxybenzyl chloride (XXIV). XXIII (30 g.) refluxed 1.5 hrs. with 45 cc. SOCl2 yielded 2-(4-

methoxybenzylthio)benzoyl chloride (XXV), m. 106-8.degree. (C6H6-petr. ether). This chloride with EtOH, as for II, gave Et 2-(4-methoxybenzylthio)benzoate, m. 100.degree.. Condensing XXIII with XXIV gave p-methoxybenzyl 2-(4-methoxybenzylthio)benzoate (XXVI), m. 114-15.degree.. XXVI, on hydrolysis, gave XXIII. NH3 in 8 cc. dioxane, treated dropwise with 3 g. XXV in 10 cc. dioxane, the mixt. kept 3 hrs. at room temp., dild. with 40 cc. H2O, neutralized with dil. HCl, and the ppt. filtered off and crystd. from EtOH yielded 2-(4-methoxybenzylthio)benzamide, m. 147.degree.. From XXV and aniline 2-(4-methoxybenzylthio)benzanilide, m. 135.degree., was obtained. Also prepd. were: 2-(4-methoxybenzylthio)-N-butylbenzamide, m. 87-90.degree.; 2-(4-methoxybenzylthio)-N-benzylbenzamide, m. 107-9.degree.; 2-(4-methoxybenzylthio)benzohydrazide (XXVIa), m. 143.degree.; 2-(4-nitrobenzylthio)benzoyl chloride, m. 128-9.degree.; Et 2-(4-nitrobenzylthio)benzoate (XXVII), m. 91.degree.; 2-(4-nitrobenzylthio)benzamide (XXVIII), m. 143-4.degree.; 2-(4-nitrobenzylthio)benzanilide (XXIX), m. 116.degree.; 2-(4-nitrobenzylthio)-N-butylbenzamide (XXX), m. 87-9.degree.; 2-(4-nitrobenzylthio)-N-benzylbenzamide (XXXI), m. 140.degree.. XXVII (1.5 g.), refluxed 1 hr. with 3 cc. 95% hydrazine and the soln. neutralized with AcOH yielded 2-(4-nitrophenyl)-3-hydroxybenzothiophene (XXXII), m. 195.degree.. XXXII was also obtained by condensing XXVII with NaOMe.. XXVII (5 g.) in 50 cc. 95% EtOH autoclaved with H at 50 atm. and 65.degree. with 0.3 g. Raney Ni 8 hrs. yielded Et 2-(4-aminobenzylthio)benzoate (XXXIII), m. 106.degree.. The acetyl deriv. (XXXIV), m. 158.degree., was obtained by refluxing XXXIII with AcOH in presence of a drop of AcCl. By this procedure, from XXVIII, 2-(4-aminobenzylthio)benzamide, m. 173.degree., was obtained; the Ac deriv., m. 262.degree., was synthesized by the same method as for XXXIV. The catalytic redn. of XXIX at 70 atm. yielded 2-(4-aminobenzylthio)benzanilide, m. 120-1.degree.; Ac deriv. m. 215.degree.. XXX and XXXI heated at 50.degree./50 atm. 5 hrs. gave 2-(4-aminobenzylthio)-N-butylbenzamide (XXXV), m. 92.degree. (Ac deriv. m. 209.degree.), and 2-(4-aminobenzylthio)-N-benzylbenzamide (XXXVI), m. 119-20.degree. (Ac deriv. m. 213.degree.). XXXIII (1 g.) in 10% dioxane with 5 g. 95% hydrazine, and the mixt. refluxed 3 hrs. gave 2-(4-aminobenzylthio)benzohydrazide, m. 197-8.degree. (EtOH). The 2-benzylthiobenzamides prepd. were tested in vitro on Candida albicans ATCC 10231 and Trichophyton mentagrophytes ATCC 8757. All the substances proved to be inactive within the limits of soly. (between 5 and 50 .gamma./cc.) or at the max. concn. of 100 .gamma./cc. against the yeast-like microorganism. Against T. mentagrophytes IX, XX, XXI, XXII, XXVIa, XXXV, and XXXVI proved to be active. The same substances were tested in vitro against Madurella grisea, Microsporum audouini, Stemphylium sarciniforme, Aspergillus fumigatus, Cryptococcus neoformans, and Nocardia asteroides and good antifungal activity was found.

IT **1531-81-3**, Benzoyl chloride, o-(benzylthio)- **92153-07-6**, Benzoyl chloride, o-(p-chlorobenzylthio)- **101094-73-9**, Benzoyl chloride, o-(p-nitrobenzylthio)- **101096-14-4**, Benzoyl chloride, o-(p-methoxybenzylthio)-
(prepn. of)

RN 1531-81-3 HCAPLUS

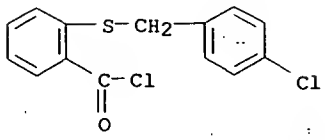
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



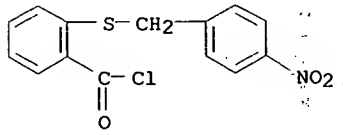
RN 92153-07-6 HCAPLUS

Searched by Barb O'Bryen, STIC 308-4291

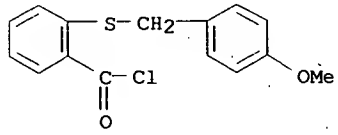
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)



RN 101094-73-9 HCAPLUS
CN Benzoyl chloride, o-(p-nitrobenzylthio)- (6CI) (CA INDEX NAME)



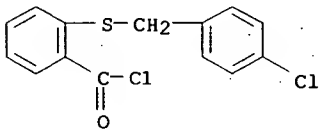
RN 101096-14-4 HCAPLUS
CN Benzoyl chloride, o-(p-methoxybenzylthio)- (6CI) (CA INDEX NAME)



L36 ANSWER 24 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1958:65715 HCAPLUS
DOCUMENT NUMBER: 52:65715
ORIGINAL REFERENCE NO.: 52:11772c-i
TITLE: Toxicity of organic sulfides to the eggs and larvae of the two-spotted spider mite. IV. Benzyl phenyl sulfides substituted by halogens and other groups
AUTHOR(S): Brookes, R. F.; Clark, N. G.; Cranham, J. E.; Greenwood, D.; Marshall, J. R.; Stevenson, H. A.
CORPORATE SOURCE: Boots Pure Drug. Co. Ltd., Nottingham, UK
SOURCE: J. Sci. Food Agr. (1958), 9, 111-15
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB cf. C.A. 25, 4543h. A series of benzyl phenyl sulfides substituted by halogens and other groups, together with some of the corresponding sulfoxides and sulfones, were characterized and their toxicities to the eggs and young of Tetranychus telarius detd. With several exceptions, the compds. were prepd. from the appropriately substituted arenethiols and benzyl halides, e.g. .omicron.-carbamoylphenyl p-chlorobenzyl sulfide was prepd. from the corresponding acid by way of .omicron.-chlorocarbonylphenyl p-chlorobenzyl sulfide, m. 108.degree.. The following XC6H4CH2SC6H4Y were prepd. (X, Y, and m.p. given): H, (4-Cl, 2-Me), 40-1.degree.; H, (4-Cl, 3-Me), 38-9.degree.; H, (5-Cl, 2-Me), 47.degree.; H, (2,4-Cl2, 3-Me), 82.degree.; H, (2,4-Cl, 5-Me), 87-8.degree.; p-F, p-Me, 61.5-2.5.degree.; p-F, p-OMe, 57.5-8.5.degree.; p-Cl, p-Me, 70.degree.; p-Cl, .omicron.-OH, - (bl 156-8.degree.); p-Cl, p-OH, 92-3.degree.; p-Cl, p-OMe, 51.degree.; p-Cl, p-OC5H11, 38.degree.;

p-Cl, p-OCH2CH2OH, 80-1.degree.; p-Cl, p-OCH2CH2SCN, 72-3.degree.; p-Cl, p-OCH2CO2H, 131-2.degree.; p-Cl, p,p'-OCH2C6H4Cl, 127.degree.; p-Cl, (4-Cl, 2-Me), 50-1.degree.; p-Cl, (4-Cl, 3:-Me), 59-60.degree.; p-Cl, (2,4-Cl2, 3-Me), 82.degree.; p-Cl, (2,4-Cl2, 5-Me), 55.degree.; p-Cl, .omicron.-CN, 55-6.degree.; p-Cl, .omicron.-CO2H, 222.degree.; p-Cl, .omicron.-CO2Me, 102.degree.; p-Cl, .omicron.-CO2Et, 87.degree.; p-Cl, .omicron.-CONH2, 144-5.degree.; p-Cl, p-Et, 66-7.degree.; 2,6-Cl2, (2,4-Cl2, 3-Me), 111-12.degree.; p-Br, p-Me, 75.degree.; p-I, p-Me, 93.degree.; p-CN, p-F, 48-9.degree.; p-CN, p-Cl, 75-7.degree.; p-Me, p-F, 44.5-5.5.degree.; p-Me, p-Cl, 80-1.degree.; p-Me, p-I, 110.degree.; p-OMe, p-F, 71.5-2.5.degree.; p-OMe, p-Cl, 80.degree.; p-OMe, p-I, 120.degree.; p-NCS, p-Cl, 80.degree.; H, (2-Cl, 5-NO2), 110-11.degree.; H, (4-Cl, 2-NO2), 129-30.degree.; .omicron.-Cl, (4-Cl, 2-NO2), 168-9.degree.; m-Cl, (2-Cl, 5-NO2), 108-19.degree.; p-Cl, p-NO2, 114-15.degree.; p-Cl, (2-Cl, 5-NO2), 153.5-4.5.degree.; p-Cl, (4-Me, 3-NO2), 64-5.degree.; p-Cl, (2-OMe, 4-NO2), 136.5-7.0.degree.; p-Me, (4-Cl, 2-NO2), 165-6.degree.; (4-OMe, 3-NO2), (4-Cl, 2-NO2), 177.0-7.5.degree.; (4-OMe, 3-NO2), p-Cl, 76-7.degree.; p-NO2, p-Cl, 66-7.degree.; p-NH2, p-Cl, 98.5-9.5.degree.; and p-NO2, (4-Cl, 2-NO2), 229-30.degree.. The following XC6H4CH2SOnC6H4Y were prepd. and tested (X, Y, n, and m.p. given): p-F, p-Me, 1, 162-3.degree.; p-F, p-Me, 2, 171-2.degree.; p-F, p-OMe, 1, 138-9.degree.; p-F, p-OMe, 2, 139-40.degree.; p-Br, p-Me, 1, 161.degree.; p-Br, p-Me, 2, 171-2.degree.; p-I, p-Me, 1, 174.degree.; p-I, p-Me, 2, 195.degree.; p-I, p-OMe, 1, 174.degree.; p-I, p-OMe, 2, 181.degree.; p-Me, p-F, 2, 140-1.degree.; p-Me, p-I, 1, 170.degree.; p-Me, p-I, 2, 172.degree.; p-OMe, p-F, 2, 167-8.degree.; p-OMe, p-Cl, 2, 153-4.degree.; p-Cl, (2-Cl, (5-NO2), 2, 181-2.degree.; p-Cl, (2-OMe, 4-NO2), 2, 162-3.degree.; (4-OMe, 3-NO2), p-Cl, 2, 166.0-6.5.degree.; (4-OMe, 3-NO2), (4-Cl, 2-NO2), 2, 152.5.degree. (decompn.); p-NO2, p-Cl, 1, 153.5-4.5.degree.; and p-NO2, p-Cl, 2, 175-6.degree.. No appreciable activity was found when the benzyl moiety was not substituted, but some compds. showed considerable activity when the nucleus of this moiety carried a p-Cl substituent. NO2, CN, and the other substituents tested had, in general, significant effects on biol. activity. None of the sulfoxides and sulfones had appreciable activity.

IT **92153-07-6**, Benzoyl chloride, o-(p-chlorobenzylthio)-
(prepn. of)
RN 92153-07-6 HCAPLUS
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)



L36 ANSWER 25 OF 32 HCAPLUS COPYRIGHT 2003 ACS
ACCESSION NUMBER: 1958:11324 HCAPLUS
DOCUMENT NUMBER: 52:11324
ORIGINAL REFERENCE NO.: 52:2069i,2070a-c
TITLE: Sulfur-containing compounds
INVENTOR(S): Stevenson, Herbert A.; Greenwood, Douglas; Higgons, Dennis J.; Cranham, John E.
PATENT ASSIGNEE(S): Boots Pure Drug Co. Ltd.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

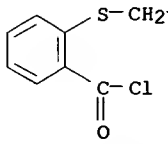
Searched by Barb O'Bryen, STIC 308-4291

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 780520		19570807	GB	

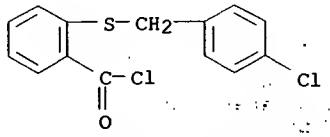
AB New benzyl phenyl sulfides have been synthesized which are valuable for the control of Tetranychidae (Red Spider mites), e.g., Tetranychus telarius L. and Metatetranychus ulmi Koch. A mixt. of 8.5 g. p-ClC6H4SH, 10 g. of p-NCC6H4CH2Br, 1.4 g. Na, and 100 cc. alc. was refluxed two hrs., cooled, and dild. with 500 cc. H2O, and the ppt. filtered off to give p-chlorophenyl p-cyanobenzyl sulfide, m. 76-7.degree. (alc.). The following compds. were prepd. in a similar way: p-cyanobenzyl phenyl sulfide (m. 73-4.degree.), p-cyanobenzyl p-fluorophenyl sulfide (m. 48-9.degree.), .omicron.-(p-cyanobenzylthio)benzoic acid (m. 220.degree.), and .omicron.-(p-chlorobenzylthio)benzoic acid (m. 222.degree.). By stirring 16.8 g. .omicron.-(p-chlorobenzylthio)benzyl chloride with 300 cc. aq. NH3, .omicron.-(p-chlorobenzylthio)benzamide, m. 144-5.degree., was prepd. .omicron.-(p-cyanobenzylthio)benzamide (m. 155-6.degree.) and .omicron.-(benzylthio)benzamide, m. 152-3.degree., were similarly prepd. A prepn. of p-chlorobenzyl .omicron.-cyanophenyl sulfide was made from 2.21 g. POCl3 in 10 cc. dry C5H5N and 2.0 g. .omicron.-(p-chlorobenzylthio)benzamide, m. 55-6.degree.. Benzyl .omicron.-cyanophenyl sulfide (m. 65-6.degree.) and p-cyanobenzyl .omicron.-cyanophenyl sulfide, m. 109-10.degree. were prepd. in the same manner. Et .omicron.-(p-chlorobenzylthio)benzoate (m. 87.degree.) was prepd. from the acid and EtOH in the presence of H2SO4. The Me ester, m. 102.degree., was prepd.

IT **1531-81-3**, Benzoyl chloride, o-(benzylthio)- **92153-07-6**, Benzoyl chloride, o-(p-chlorobenzylthio)- **100965-29-5**, Benzoyl chloride, o-(p-cyanobenzylthio)-
(prepn. of)

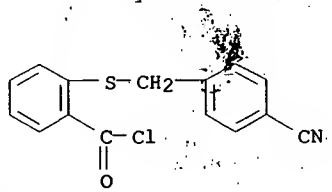
RN 1531-81-3 HCAPLUS
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



RN 92153-07-6 HCAPLUS
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)



RN 100965-29-5 HCAPLUS
CN Benzoyl chloride, o-(p-cyanobenzylthio)- (6CI) (CA INDEX NAME)



Searched by Barb O'Bryen, STIC 308-4291

L36 ANSWER 26 OF 32 USPATFULL
ACCESSION NUMBER: 97:1479 USPATFULL
TITLE: Organic nitrates, processes for their preparation and their use in the treatment of cardiovascular diseases
INVENTOR(S): Nallet, Jean-Pierre, Montaney, France
Dreux, Jacques, Lyons, France
Berdeaux, Alain, Paris, France
Richard, Vincent, Paris, France
Martorana, Piero, Bad Homburg, Germany, Federal Republic of
Bohn, Helmut, Schoneck, Germany, Federal Republic of
PATENT ASSIGNEE(S): Laboratoires Hoechst, SA, Puteaux, France (non-U.S. corporation)

	NUMBER	KIND	DATE	
PATENT INFORMATION:	US 5591758		19970107	
	WO 9303037		19930218	
APPLICATION INFO.:	US 1993-971812		19930504	(7)
	WO 1992-EP1746		19920801	
			19930504	PCT 371 date
			19930504	PCT 102(e) date

	NUMBER	DATE
PRIORITY INFORMATION:	FR 1991-10039	19910807

DOCUMENT TYPE: Utility
FILE SEGMENT: Granted
PRIMARY EXAMINER: Gerstl, Robert
LEGAL REPRESENTATIVE: Perman & Green
NUMBER OF CLAIMS: 7
EXEMPLARY CLAIM: 1
NUMBER OF DRAWINGS: 7 Drawing Figure(s); 2 Drawing Page(s)
LINE COUNT: 2275
CAS INDEXING IS AVAILABLE FOR THIS PATENT.

AB Organic nitrates, processes for their preparation and their use in the treatment of vascular diseases and in particular in the treatment of angina.

The said nitrates correspond to the following formula I:

R--CO--(A).sub.n --Y--B (I)

in which:

R represents, in particular, a sulphur-containing radical and a sulphur-containing amino acid residue; A represents, in particular, a CH.sub.2 group or a substituted amino acid; n is 0 or 1 or greater than 1; Y represents an oxygen atom or an NH group and B represents, in particular, a 1,4:3,6-dianhydro hexitol mononitrate radical, an itol nitrate radical or an inositol radical.

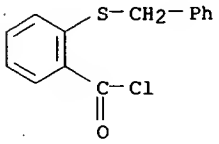
The said organic nitrates are prepared by reacting:

I. either a thio acid of the type R--COOH, in which R has the same meaning as above, with a derivative of formula II: (A).sub.n --Y--B, in which A, Y, B and n have the same meaning as above,

II. or a derivative of formula III: R--CO--(A).sub.n, in which R, A and n have the same meaning as above, with a derivative of formula Y--B, in which Y and B have the same meaning as above, in an appropriate solvent

and under non-epimerising conditions.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **1531-81-3**, S-Benzylthiosalicylic acid chloride
(esterification and amidation of, in prepn. of vasorelaxants)
RN 1531-81-3 USPATFULL
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 27 OF 32 USPATFULL
ACCESSION NUMBER: 94:30826 USPATFULL
TITLE: Chelating agents for forming complexes with radioactive isotopes, metal complexes thereof and use thereof in diagnosis and therapy
INVENTOR(S): Neumeier, Reinhard, Berlin, Germany, Federal Republic of
Kramp, Wolfgang, Berlin, Germany, Federal Republic of
Macke, Helmut R., Lorrach, Germany, Federal Republic of
PATENT ASSIGNEE(S): Institut fur Diagnostikforschung GmbH, Berlin, Germany, Federal Republic of (non-U.S. corporation)

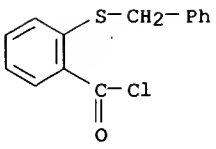
	NUMBER	KIND	DATE
PATENT INFORMATION:	US 5302370		19940412
APPLICATION INFO.:	US 1990-572140		19900822 (7)

	NUMBER	DATE
PRIORITY INFORMATION:	DE 1989-3930674	19890911

DOCUMENT TYPE: Utility
FILE SEGMENT: Granted
PRIMARY EXAMINER: Stoll, Robert L.
ASSISTANT EXAMINER: Covert, John M.
LEGAL REPRESENTATIVE: Millen, White, Zelano & Branigan
NUMBER OF CLAIMS: 8
EXEMPLARY CLAIM: 1
LINE COUNT: 1375
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB The invention relates to compounds having the general formula I ##STR1## where A if required can contain a functional and/or activated group C for coupling to selectively concentrating compounds or can contain a selectively concentrating compound coupled via the group C. B and B' are functional groups for coordinate bonding of groups carrying metal ions. The novel compounds are for forming complexes with radioactive metal ions, more particularly rhenium and technetium isotopes, and are used in medical diagnosis and therapy.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **1531-81-3**, S-Benzylthiosalicylic acid chloride
(acylation by, of propanediamine deriv.; in prepn. of bidentate ligands)
RN 1531-81-3 USPATFULL
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

Searched by Barb O'Bryen, STIC 308-4291

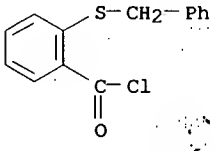


L36 ANSWER 28 OF 32 USPATFULL
ACCESSION NUMBER: 91:16809 USPATFULL
TITLE: Herbicidal sulfonamides
INVENTOR(S): Rorer, Morris P., Newark, DE, United States
PATENT ASSIGNEE(S): E. I. du Pont de Nemours and Company, Wilmington, DE, United States (U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 4995901		19910226
APPLICATION INFO.:	US 1990-461581		19900105 (7)

RELATED APPLN. INFO.: Division of Ser. No. US 1988-204556, filed on 15 Jun 1988, now patented, Pat. No. US 4906282 which is a continuation-in-part of Ser. No. US 1987-78191, filed on 27 Jul 1987, now abandoned
DOCUMENT TYPE: Utility
FILE SEGMENT: Granted
PRIMARY EXAMINER: Ford, John M.
LEGAL REPRESENTATIVE: Costello, James A.
NUMBER OF CLAIMS: 22
EXEMPLARY CLAIM: 1,21
LINE COUNT: 4390
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB Herbicidal sulfonamides having the general formula ##STR1## wherein J, W, R and A are more particularly described herein, such compounds and agricultural compositions containing them being useful as preemergent or postemergent herbicides or both, or as plant growth regulants, including the manner of their use.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **1531-81-3P**
(prepn. and reaction of, with methoxylamine)
RN 1531-81-3 USPATFULL
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 29 OF 32 USPATFULL
ACCESSION NUMBER: 90:17333 USPATFULL
TITLE: Herbicidal sulfonamides
INVENTOR(S): Rorer, Morris P., Newark, DE, United States
PATENT ASSIGNEE(S): E. I. Du Pont de Nemours and Company, Wilmington, DE, United States (U.S. corporation)

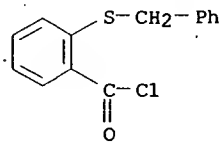
	NUMBER	KIND	DATE

Searched by Barb O'Bryen, STIC 308-4291

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 4906282		19900306
APPLICATION INFO.:	US 1988-204556		19880615 (7)

RELATED APPLN. INFO.: Continuation-in-part of Ser. No. US 1987-78191, filed on 27 Jul 1987, now abandoned
DOCUMENT TYPE: Utility
FILE SEGMENT: Granted
PRIMARY EXAMINER: Ford, John M.
LEGAL REPRESENTATIVE: Costello, James A.
NUMBER OF CLAIMS: 23
EXEMPLARY CLAIM: 1,22
LINE COUNT: 4364
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB Herbicidal sulfonamides having the general formula ##STR1## wherein J, W, R and A are more particularly described herein, such compounds and agricultural compositions containing them being useful as preemergent or postemergent herbicides or both, or as plant growth regulants, including the manner of their use.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **1531-81-3P**
(prepn. and reaction of, with methoxylamine)
RN 1531-81-3 USPATFULL
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



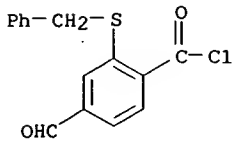
L36 ANSWER 30 OF 32 USPATFULL
ACCESSION NUMBER: 78:941 USPATFULL
TITLE: 6,11-Dihydrodibenzo-[b. e.]-thiepin-11-one-3-aldehyde and 3-acetal derivatives
INVENTOR(S): Ackrell, Jack, Palo Alto, CA, United States
PATENT ASSIGNEE(S): Syntex (U.S.A.) Inc., Palo Alto, CA, United States (U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 4066663		19780103
APPLICATION INFO.:	US 1976-701780		19760701 (5)

DOCUMENT TYPE: Utility
FILE SEGMENT: Granted
PRIMARY EXAMINER: Jaisle, Cecilia M. S.
LEGAL REPRESENTATIVE: Blaufarb, Gerard A., Walker, William B.
NUMBER OF CLAIMS: 18
EXEMPLARY CLAIM: 1,14
LINE COUNT: 691
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB The novel compounds 6,11-dihydrodibenzo[b.e.]-thiepin-11-one-3-acetaldehyde and (dl) 2(6,11-dihydrodibenzo-[b.e.]-thiepin-11-one-3-yl)propionaldehyde, certain dialkyl- and cyclic acetals thereof, and processes and novel intermediates for making same.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **64976-84-7**
(pren. and cyclization of)

RN 64976-84-7 USPATFULL
CN Benzoyl chloride, 4-formyl-2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

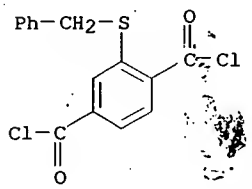


L36 ANSWER 31 OF 32 USPATFULL
ACCESSION NUMBER: 76:70718 USPATFULL
TITLE: 6,11-Dihydrodibenzo-thiepin-11-ones, compositions and uses thereof
INVENTOR(S): Ackrell, Jack, Mexico City, Mexico
PATENT ASSIGNEE(S): Syntex (U.S.A.) Inc., Palo Alto, CA, United States (U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 4000308		19761228
APPLICATION INFO.:	US 1975-634086		19751121 (5)

RELATED APPLN. INFO.: Continuation-in-part of Ser. No. US 1975-550316, filed on 18 Feb 1975, now abandoned And Ser. No. US 1975-591725, filed on 30 Jun 1975, now abandoned , said Ser. No. 591725 which is a continuation-in-part of Ser. No. 550316
DOCUMENT TYPE: Utility
FILE SEGMENT: Granted
PRIMARY EXAMINER: Jiles, Henry R.
ASSISTANT EXAMINER: Jaisle, C. M. S.
LEGAL REPRESENTATIVE: Blaufarb, Gerard A., Walker, William B.
NUMBER OF CLAIMS: 12
EXEMPLARY CLAIM: 1
LINE COUNT: 1425
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB This invention relates to novel 6,11-dihydrodibenzo[b.e.]-thiepin-11-ones, methods of preparation, compositions and uses thereof.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **61220-65-3P**
(prepn. and cyclization of)
RN 61220-65-3 USPATFULL
CN 1,4-Benzenedicarbonyl dichloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L36 ANSWER 32 OF 32 USPATFULL
ACCESSION NUMBER: 76:70698 USPATFULL

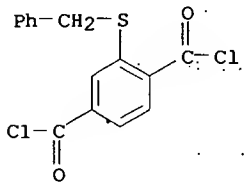
Searched by Barb O'Bryen, STIC, 308-4291

TITLE: 6,11-Dihydrodibenzo-thiepin-11-ones, compositions and uses thereof
INVENTOR(S): Ackrell, Jack, Mexico City, Mexico
PATENT ASSIGNEE(S): Syntex (U.S.A.) Inc., Palo Alto, CA, United States (U.S. corporation)

	NUMBER	KIND	DATE
PATENT INFORMATION:	US 4000288		19761228
APPLICATION INFO.:	US 1975-634085		19751121 (5)

RELATED APPLN. INFO.: Continuation-in-part of Ser. No. US 1975-550316, filed on 18 Feb 1975, now abandoned And Ser. No. US 1975-591725, filed on 30 Jun 1975, now abandoned , said Ser. No. 591725 which is a continuation-in-part of Ser. No. 550316
DOCUMENT TYPE: Utility
FILE SEGMENT: Granted
PRIMARY EXAMINER: Jiles, Henry R.
ASSISTANT EXAMINER: Jaisle, C. M. S.
LEGAL REPRESENTATIVE: Walker, William B., Blaufarb, Gerard A.
NUMBER OF CLAIMS: 26
EXEMPLARY CLAIM: 1
LINE COUNT: 1472
CAS INDEXING IS AVAILABLE FOR THIS PATENT.
AB This invention relates to novel 6,11-dihydrodibenzo-[b.e.]-thiepin-11-ones, methods of preparation, compositions and uses thereof.

CAS INDEXING IS AVAILABLE FOR THIS PATENT.
IT **61220-65-3P**
(prepn. and cyclization of)
RN 61220-65-3 USPATFULL
CN 1,4-Benzenedicarbonyl dichloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



[redacted] ENTERED AT 12:36:11 ON 09 APR 2003
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.

FILE COVERS 1907-1966
FILE LAST UPDATED: 01 May 1997 (19970501/UP)

This file contains CAS Registry Numbers for easy and accurate substance identification. Title keywords, authors, patent assignees, and patent information, e.g., patent numbers, are now searchable from 1907-1966. TIFF images of CA abstracts printed between 1907-1966 are available in the PAGE display formats.

Searched by Barb O'Bryen, STIC 308-4291

This file supports REGISTRY for direct browsing and searching of all substance data from the REGISTRY file. Enter HELP FIRST for more information.

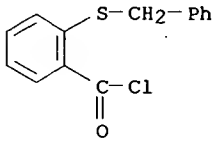
L25 STR
L28 29 SEA FILE=REGISTRY SSS FUL L25
[redacted]

[redacted] fil hom

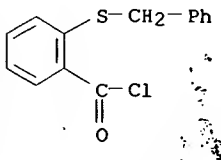
L32 ANSWER 1 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA59:10010f CAOLD
TITLE: 11-(3-dimethylaminopropylidene)-6,11-dihydrodibenz(b,e)thiepin
AUTHOR NAME: Protiva, Miroslav; Rajsner, M.; Votava, Z.; Metysova, J.
DOCUMENT TYPE: Patent

PATENT NO.	KIND	DATE
PI CZ 105590		

INDEX TERM: 1531-77-7 **1531-81-3** 1531-85-7 96175-10-9
IT **1531-81-3**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



L32 ANSWER 2 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA59:2772g CAOLD
TITLE: synthetic ataractics - (VII) 11-(3-dimethylaminopropylidene)-6,11-dihydrodibenzo[b,e]thiepins
AUTHOR NAME: Rajsner, Miroslav; Protiva, M.
INDEX TERM: 113-53-1 897-15-4 1531-77-7 **1531-81-3** 1531-85-7 1699-03-2 1699-04-3 1745-46-6 33301-21-2 34129-26-5 96175-10-9
IT **1531-81-3**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

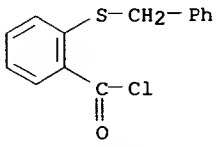


L32 ANSWER 3 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA58:4574c CAOLD
TITLE: synthetic medicinals - (VIII) tricyclic thiazepine and thiepin derivs.
AUTHOR NAME: Gadient, Fulvio; Jucker, E.; Lindenmann, A.; Taeschler, M.

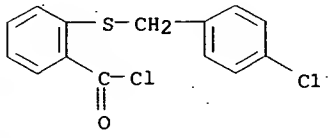
Searched by Barb O'Bryen, STIC 308-4291

INDEX TERM: 113-53-1 825-83-2 852-23-3 1152-03-0 1152-04-1
1531-77-7 1531-78-8 1531-79-9 **1531-81-3**
1531-82-4 1531-83-5 1531-85-7 1531-87-9 1531-88-0
1531-89-1 1531-91-5 1531-92-6 1662-82-4 1662-83-5
1662-84-6 1662-85-7 1662-87-9 1699-03-2 1702-31-4
1705-49-3 2647-35-0 2796-88-5 2991-42-6 4504-99-8
4673-21-6 4677-29-6 4683-76-5 5201-79-6 5202-02-8
5202-03-9 5202-05-1 5202-07-3 5202-08-4 5202-09-5
5202-10-8 5202-11-9 5500-40-3 13448-33-4 20979-33-3
23772-04-5 73150-00-2 73150-01-3 82401-08-9 89581-84-0
92153-07-6 92696-10-1 93698-31-8 93698-32-9
94911-40-7 95227-39-7 95424-20-7 96214-79-8 96674-50-9
96982-32-0 97254-84-7 97254-90-5 97255-01-1 100152-58-7
100196-55-2 100211-69-6 100323-31-7 100627-35-8 100771-20-8
100771-22-0 101231-50-9 101319-05-5 103534-81-2 103908-42-5
103908-43-6 107204-82-0

IT **1531-81-3** **92153-07-6**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



RN 92153-07-6 CAOLD
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)

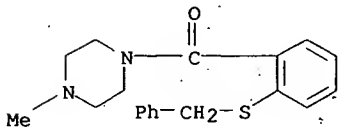


L32 ANSWER 4 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA56:4664g CAOLD
TITLE: dialkylaminoalkylic N-or S-derivs. of 2-mercapto-2,2'-dithio, 2-(alkylthio)-, 2-(aralkylthio)-, and 2-(arylthio)benzamides
AUTHOR NAME: Chiaidi, Franco; Ponci, R.; Baruffini, A.
INDEX TERM: 1049-92-9 2634-31-3 2752-93-4 15109-12-3 20904-30-7
32276-24-7 32276-25-8 32276-26-9 72534-70-4 88783-54-4
90793-61-6 90919-33-8 91061-47-1 91430-12-5 91767-36-1
91822-89-8 92199-75-2 92374-01-1 93010-85-6 93994-99-1
94032-03-8 94208-07-8 94262-71-2 94326-49-5 94378-58-2
94437-14-6 94437-53-3 94682-59-4 94758-14-2 94862-94-9
94906-16-8 94907-25-2 94915-86-3 94999-40-3 95277-72-8
95291-17-1 96063-90-0 96067-38-8 96198-56-0 97018-37-6
97393-84-5 97575-12-7 97772-27-5 98051-88-8 98131-92-1
98200-27-2 98397-89-8 98470-98-5 98766-48-4
98883-91-1 **98963-55-4** 99003-05-1
99729-67-6 100027-88-1 100197-42-0 100233-06-5 100321-14-0
103133-24-0 103193-14-2 **103193-31-3** 107305-87-3
107579-58-8 108042-03-1

IT **98883-91-1** **98963-55-4** **103193-31-3**

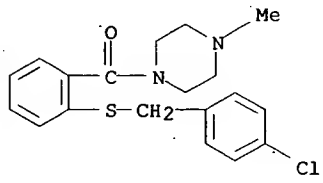
Searched by Barb O'Bryen, STIC 308-4291

RN 98883-91-1 CAOLD
CN Piperazine, 1-[o-(benzylthio)benzoyl]-4-methyl-, hydrochloride (7CI) (CA INDEX NAME)



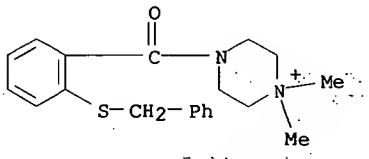
● HCl

RN 98963-55-4 CAOLD
CN Piperazine, 1-[o-[(p-chlorobenzyl)thio]benzoyl]-4-methyl-, hydrochloride (7CI) (CA INDEX NAME)



● HCl

RN 103193-31-3 CAOLD
CN 4-[o-(Benzylthio)benzoyl]-1,1-dimethylpiperazinium iodide (7CI) (CA INDEX NAME)



● I⁻

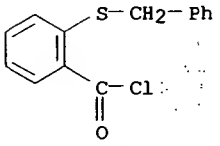
L32 ANSWER 5 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA55:21040b CAOLD
TITLE: 2-benzylthiobenzamides with antifungal activity
AUTHOR NAME: Gialdi, Franco; Ponci, R.; Baruffini, A.
INDEX TERM: 791-31-1 824-94-2 1485-70-7 1531-80-2
1531-81-3 2527-62-0 13156-90-6 15887-84-0
51471-69-3 54705-18-9 58435-43-1 **92153-07-6**
100073-03-8 100542-71-0 100714-50-9 100716-36-7 100870-00-6

Searched by Barb O'Bryen, STIC 308-4291

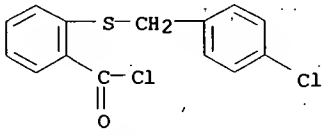
100873-51-6 101090-02-2 101093-83-8 **101094-73-9**
101096-13-3 **101096-14-4** 101112-67-8 101280-27-7
101282-64-8 101284-41-7 101293-51-0 101595-21-5 101718-04-1
101718-05-2 101729-82-2 101730-14-7 101784-06-9 101784-07-0
101785-12-0 101792-10-3 102002-72-2 102007-29-4 102010-53-7
102016-74-0 102080-50-2 102241-14-5 102311-03-5 102311-66-0
102318-18-3 102441-66-7 102456-84-8 102457-56-7 102459-58-5
102462-88-4 102478-82-0 102479-03-8 102889-63-4 106472-86-0
108844-43-5 109938-67-2 110436-90-3 112152-55-3 112690-22-9

IT **1531-81-3 92153-07-6 101094-73-9**
101096-14-4

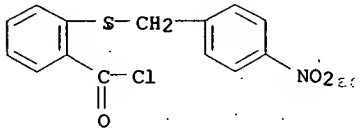
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)



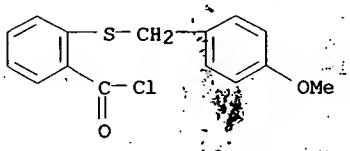
RN 92153-07-6 CAOLD
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)



RN 101094-73-9 CAOLD
CN Benzoyl chloride, o-(p-nitrobenzylthio)- (6CI) (CA INDEX NAME)



RN 101096-14-4 CAOLD
CN Benzoyl chloride, o-(p-methoxybenzylthio)- (6CI) (CA INDEX NAME)



L32 ANSWER 6 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA52:11772c CAOLD
TITLE: stereochemistry of base-catalyzed addns. of p-toluenethiol

Searched by Barb O'Bryen, STIC 308-4291

to negatively-substituted acetylenes - (II) kinetics of the reaction between Na p-toluenethiolate and phenylacetylene, (III) aryl ethynyl sulfone, (IV) isolation of an intermediate in the base-catalyzed reaction of p-toluenethiol with tetrachloroethene

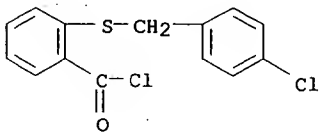
AUTHOR NAME: Heine, Richard F.

TITLE: toxicity of org. sulfides to the eggs and larvae of the two-spotted spider mite - (IV) benzyl phenyl sulfides substituted by halogens and other groups

AUTHOR NAME: Brookes, Robert F.; Clark, N. G.; Cranham, J. E.; Greenwood, D.; Marshall, J. R.; Stevenson, H. A.

INDEX TERM: 726-39-6 1426-51-3 1426-52-4 1426-53-5 1494-29-7
1494-30-0 1494-33-3 1494-34-4 2966-00-9 2966-01-0
5023-72-3 6969-14-8 15887-84-0 17530-85-7 22057-45-0
26885-97-2 41866-56-2 83582-88-1 84035-83-6 87740-12-3
88275-95-0 **92153-07-6** 95309-86-7 96459-88-0
96460-19-4 99513-98-1 99514-48-4 99514-49-5 99514-50-8
100398-52-5 100542-71-0 100542-79-8 100542-85-6 100542-92-5
100542-93-6 100622-88-6 100622-92-2 100716-17-4 100716-25-4
100716-68-5 100716-85-6 100717-12-2 100717-13-3 100717-14-4
100717-15-5 100717-16-6 100717-17-7 100717-18-8 100717-20-2
100717-23-5 100717-24-6 101094-78-4 101094-80-8 101094-82-0
101096-13-3 101096-24-6 101118-82-5 101282-56-8 101282-64-8
101353-13-3 101353-27-9 103206-26-4 105946-59-6 106037-73-4
106737-65-9 107776-80-7 107920-69-4 107920-70-7 107921-84-6
107921-85-7 108749-77-5 109038-98-4 109038-99-5

IT **92153-07-6**
RN 92153-07-6 CAOLD
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)



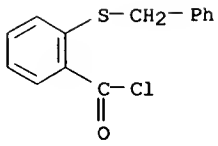
L32 ANSWER 7 OF 7 CAOLD COPYRIGHT 2003 ACS
ACCESSION NUMBER: CA52:2069i CAOLD
TITLE: S-contg. compds.
AUTHOR NAME: Stevenson, Herbert A.; Greenwood, D.; Higgons, D. J.; Cranham, J. E.
DOCUMENT TYPE: Patent
TITLE: sulfur-contg. compds.
PATENT ASSIGNEE: Boots Pure Drug Co. Ltd.
DOCUMENT TYPE: Patent

PATENT NO.	KIND	DATE
PI GB 780520		

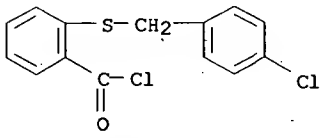
INDEX TERM: 726-39-6 **1531-81-3** 15887-84-0 51229-54-0
54705-18-9 63216-04-6 **92153-07-6** 100542-71-0
100880-37-3 100961-52-2 **100965-29-5** 100966-11-8
101094-78-4 101094-80-8 101096-13-3 101282-64-8

IT **1531-81-3 92153-07-6 100965-29-5**
RN 1531-81-3 CAOLD
CN Benzoyl chloride, 2-[(phenylmethyl)thio]- (9CI) (CA INDEX NAME)

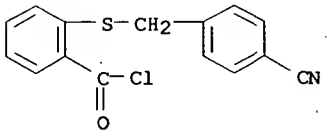
Searched by Barb O'Bryen, STIC 308-4291



RN 92153-07-6 CAOLD
CN Benzoyl chloride, o-[(p-chlorobenzyl)thio]- (6CI, 7CI) (CA INDEX NAME)



RN 100965-29-5 CAOLD
CN Benzoyl chloride, o-(p-cyanobenzylthio)- (6CI) (CA INDEX NAME)



FILE 'HOME' ENTERED AT 12:36:22 ON 09 APR 2003

Searched by Barb O'Bryen, STIC 308-4291